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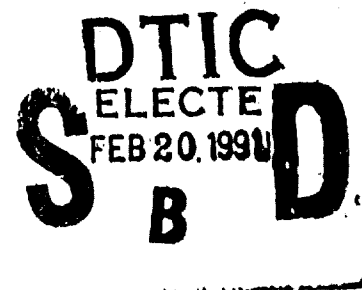
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# A PROPOSED STANDARD PRACTICE FOR FRACTOGRAPHIC ANALYSIS OF MONOLITHIC ADVANCED CERAMICS

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November 1990

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ABSTRACT

The strength of many brittle ceramics reflects the flaws present in the material and the intrinsic fracture toughness. Interpretation of strength-test results for monolithic, brittle advanced ceramics requires fractographic analysis whether mechanical testing has been done for quality control, materials development, or design purposes.

Progress is presented on an effort to develop a standard practice for fractographic analysis of laboratory strength-test specimens. The primary goal is to encourage fractography as a complement to mechanical testing. Procedures will be developed with regard to inspection techniques, sampling criteria and reporting practices. The standard practice is in a formative state and the purpose of this report is to present its framework and solicit recommendations and comments.

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## INTRODUCTION

It is not difficult to argue that fractography is essential to properly interpret mechanical strength-test results. Stressed monolithic ceramics and "simple" composite ceramics (e.g., particulate or whisker reinforced) will typically fail in a brittle fashion due to the unstable propagation of cracks from preexisting defects.\* These defects may be intrinsic to the material as a consequence of its manufacturing process, or may be introduced as a result of specimen preparation, handling, or exposure. Strength-test results must be interpreted in the context of these defects whether the goal of the mechanical testing is materials characterization or design. This was discussed in the context of flexure testing in the paper at the First Conference on Fractography of Glasses and Ceramics.<sup>1</sup> In that paper, an example from a comparative machining study was given where a completely erroneous conclusion could have been drawn from the strength data alone. Only with comprehensive fractography was it realized that the apparent differences in strength in separate samples were due to subtle flaw variations between different billets of the alumina ceramic.

The matter is even more critical if strength is to be used for design. Characterization of multiple flaw populations is important to the extent that if more than two flaw populations are present, design with the material may be difficult or even impossible.<sup>1</sup> The subtle variation in porosity defects in the comparative machining study also poses troubling questions. Defects in that study manifested themselves either as discrete voids or as zones of microporosity. Can these be considered members of a single family of defects or are they different? Thus, characterization of defects is a critical topic.

The presentation at the first fractography conference foreshadowed our effort to develop a standard practice.<sup>1</sup> Optical and scanning electron microscopy (SEM) were discussed in the context of finding and characterizing strength-limiting defects. Montages and labeled Weibull graphs were suggested schemes to aid in the interpretation and reporting of fractographic results. A possible flaw nomenclature was proposed. Two recent mechanical property round-robins have highlighted some of the shortcomings of fractographic procedures now in use.<sup>2,3</sup> An earlier exercise similarly uncovered serious interpretation problems.<sup>4</sup> It is evident that there are widely divergent practices and expertise levels.

Unfortunately, fractography is highly interpretive, and a considerable amount of judgement has to be employed. There are a number of "tricks of the trade," and fractography is a continual learning experience. New tools and analyses are continually becoming available. Success in finding and correctly characterizing defects is a strong function of the experience level of the fractographer. Even experts can be misled in their findings.<sup>2</sup> It is extremely rare to find open debate on fractographic interpretations.<sup>5,6</sup> Fractographic analysis and "representative" photographs are usually taken at face value. Most fractographers must admit that, in truth, there often are disagreements in interpretation.

\*"Flaws" and "defects" are used synonymously in this paper.

1. QUINN, G. D. *Fractographic Analysis and the Army Flexure Test Method* in *Fractography of Glasses and Ceramics*. Advances in Ceramics, Vol. 22, V. Frechette and J. Varner, eds., American Ceramic Society, Westerville, Ohio, 1988, p. 319-333.
2. QUINN, G. D. *Flexure Strength of Advanced Ceramics - A Round Robin Exercise*. U. S. Army Materials Technology Laboratory, Watertown, MA, MTL TR 89-62, July 1989.
3. FERBER, M. K., and TENNERY, V. J. *Fractographic Study of a Silicon Nitride Ceramic* in *Proceedings of the First European Ceramic Society Conference*. G. deWith, R. A. Terpstra, R. Metselaar, eds., Maastricht, NL., Elsevier, June 1989, p. 18-23.
4. LEWIS, D., III. Private Communication, 1989.
5. PURSLOW, D. *Comment on Fractography of Unidirectional Graphite-Epoxy as a Function of Moisture, Temperature and Specimen Quality*. J. Mat. Sci. Let., v. 8, 1989, p. 617.
6. CLEMENTS, L. L. *Reply to Comment on Fractography of Unidirectional Graphite-Epoxy as a Function of Moisture, Temperature and Specimen Quality*. J. Mat. Sci. Let., v. 8, 1989, p. 618.

Our objective is to advance the state-of-the-art of fractographic analysis of advanced ceramics by developing a fractographic standard practice in order to complement and enhance mechanical strength results. Indeed, designers who wish to use advanced ceramics urge such a practice as they seek more specific characterization of strength-limiting defects; however, the draft standard practice is not limited to design applications. Different sampling criteria and analyses are required depending upon the application of the fractographic data. Our goal is also to bring together the best available information on defect characterization and, by a tutorial approach, introduce newcomers to the field.

The approach of the standard practice will be to guide fractographic analysis and to present helpful tips. It will not be autocractic but, instead, will recommend sound procedures which we hope will lead to improved fractography, and fractography that will have optimum complementary value to mechanical strength results. Primary emphasis will be on location and characterization of strength-limiting defects in laboratory test specimens, but the same principles are easily adaptable to component failure analysis.

### **THE STANDARD PRACTICE: AN OUTLINE**

Table 1 gives an outline of the draft standard procedure, which will be written in a conventional format for U.S. Army Military Standards and American Society of Testing and Materials Standards. The standard will be prepared first as a U.S. Army Military Standard. The "Scope" and "Significance and Use" sections will specify that the standard is applicable to monolithic, and particle or whisker-reinforced ceramic mechanical test specimens. The objective is quite focused: find and characterize strength-limiting defects. The fractographic analysis will be for purposes of quality control, materials research and development, and design. The intended use of the results will in turn influence the extent of the fractographic analysis, as will be discussed below.

The standard cannot (and should not) delve into excessive detail on all the nuances of fractographic analysis and, therefore, we will include a detailed and annotated bibliography which will hopefully serve both newcomers and experts. A brief listing of the bibliography without annotations is provided in Appendix 1 of this paper.

### **EQUIPMENT FOR THE STANDARD PRACTICE**

The Standard will note that the following equipment is necessary:

- Binocular stereomicroscope with adjustable magnification between approximately 10X to 100X and a directional light source.
- Cleaning and preparation equipment such as a sonic bath and diamond cut-off wheel.
- SEM with energy or wavelength dispersive spectroscopy analyzer.
- Macrophotography camera stand (optional).
- Various and sundry peripheral equipment such as canned compressed air, tweezers, and holders.

Table 1. OUTLINE OF THE STANDARD PRACTICE

Title
1. Scope
2. Significance and Use
3. Summary of Practice
4. Equipment
5. Procedure
5.1 General
5.2 Mechanical Testing
5.3 Handling, Storage (Contaminants Appendix)
5.4 Visual Inspection (1X to 2X)
5.5 Optical Microscopy (10X to 100X)
5.6 Scanning Electron Microscopy (10X to 2000X)
5.7 Recording Results
6. Flaw Characterization
6.1 General
6.2 Identity --- (Nomenclature and Figure Appendix)
6.3 Location
6.4 Size
7. Report
Appendix 1. Contaminants
Appendix 2. Fracture Patterns in Mechanical Test Specimens
Appendix 3. Bibliography
Appendix 4. Suggested Flaw Nomenclature for Structural Ceramics
Appendix 5. Defect Encyclopedia
Appendix 6. Fractographic - Montages

## PROCEDURE OF THE STANDARD PRACTICE

### General

The standard practice procedure will be directed at preserving and finding the primary fracture surface and the strength-limiting defect.

Location, identification, and characterization of strength-limiting defects in advanced ceramics can sometimes be accomplished by simple visual and optical techniques, although it more often requires SEM. It is emphasized that SEM by itself is often not adequate and that optimum analysis requires both optical microscopy and SEM. To optimize a fractographic exercise, care must be taken in all steps starting prior to the mechanical testing of the specimen or component.

Sampling criteria must be established for each step of the analysis. This of course depends upon a number of factors such as: the material's conduciveness to fractography, the purpose of the exercise, and the resources available. One suggested set of sampling criteria is given in Table 2.



Table 2. SUGGESTED SAMPLING GUIDELINES

	1X Visual	10X to 100X Optical	10X to 2000X SEM
Level 1 - Quality control	Specimens which fail to meet minimum strength requirements	Specimens which fail to meet minimum strength requirements	Optional
Level 2 Screening - Quality control	All specimens	All specimens (both halves)	Optional
Level 3 Intermediate - Quality control - Materials development	All specimens	All specimens	Representative specimens (mount both halves) - 2 each of each flaw type - the 5 lowest strength specimens - at least 2 optically unidentifiable flaws
Level 4 Comprehensive - Quality control - Materials research - Design	All specimens	All specimens	All specimens, or as many specimens as necessary such that combined optical microscopy and SEM characterize 90% of all identifiable origins

Reference 2 gives an instance where sampling criteria was discussed in some detail. For that exercise, in which a sintered high-purity and a high-density alumina and reaction-bonded silicon nitride were examined, it was necessary to examine all specimens optically and approximately 20% by SEM. Preliminary assessments from optical microscopy were occasionally misleading or wrong. This usually would be detected during SEM examination. The accuracy of an optical assessment depends upon a number of factors including:

- Operator experience
- Operator patience and care
- Material suitability and conduciveness to analysis
- Defect characteristics such as size, shape, and contrast
- Equipment quality
- Lighting
- Luck

It may be somehow necessary to assign a confidence factor to the characterization of defects. SEM work could be used to verify the optical work, or to increase its confidence. Even SEM examinations is not foolproof, as it is oblivious to color and reflectivity, and cannot detect some features in transparent or translucent materials.

## Mechanical Testing

A few simple precautions should be taken prior to breaking a specimen. Markings of some sort should be placed on the specimen to maintain a point of reference. This will aid reconstruction and assessment of whether misalignments or test jig errors caused invalid fractures. Buffering material should be placed around the specimen in order to reduce secondary failures and impact damage, which can confuse and delay identification of the primary fracture surfaces or damage them.

## Handling and Storage

The testing should be done in a clean environment, and the broken pieces should be handled and stored to minimize contamination. The standard will suggest several storage media including paper envelopes, plastic or glass vials, or plastic trays. Table 3 represents advantages and disadvantages of each storage media. Masking tape can be handy in preserving specimens in a reconstructed state, but the adhesive can smear on the fracture surface and only a solvent will remove it effectively. Mounting clay **should not** be used, since clay particles are very difficult to remove and can appear quite convincingly as defects on fracture surfaces.

Table 3. COMMONLY USED STORAGE MEDIA FOR FRACTURED SPECIMENS

Storage Media	Advantage	Disadvantage
Envelopes	Notes written on, minimal space required, inexpensive	Lint contamination, specimen free to move
Glass vials	Very clean	Hard surface could cause secondary fracture, specimen free to move, expensive
Plastic trays	Clean, inexpensive saves space	Plastic contamination, specimen free to move
Tape	Inexpensive, mark primary fracture, maintain reconstructed specimen	Adhesive contamination

## Visual Examination (1X to 2X)

A visual examination under ambient light is quite beneficial and integral to normal specimen reconstruction. Overall crack patterns can be helpful in finding the primary fracture surfaces, fracture mirrors, origins, and in assessing the stress state in the specimen. Branching angles can suggest uniaxial or multiaxial stress states. Misalignments in testing can be detected, for example, when there is excessive breakage under load pins in flexure testing. The standard will contain an appendix which has schematics of typical crack patterns in common test specimens. Figure 1 is an example showing patterns in flexure bars.

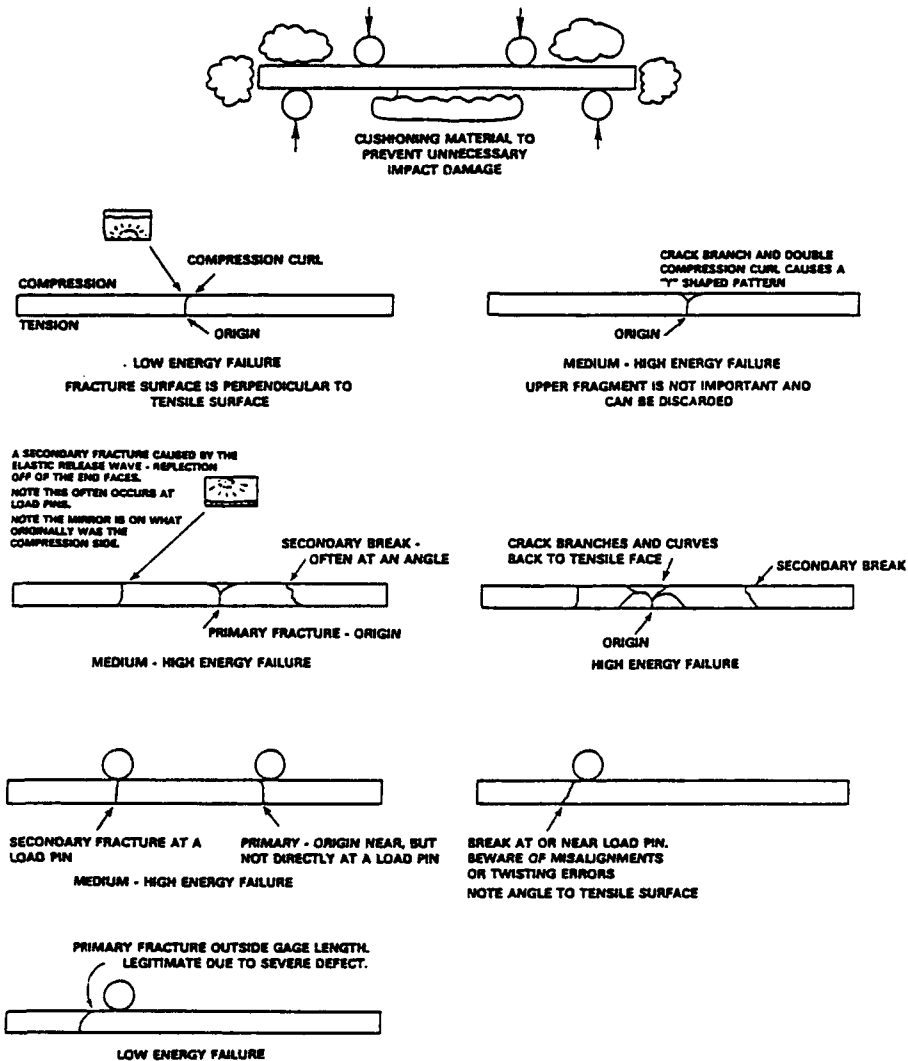


Figure 1. Fracture patterns in flexure specimens.

### Optical Examinations (10X to 100X)

Optical microscopy is best done with a stereo binocular microscope with variable magnification because it has good depth of field. A traversing stage coupled with cross hairs or a graduated reticule in the eyepiece are useful in measuring the size/area of the flaw and/or mirror. There should be a directional light source with adjustable angle of incidence onto fracture surfaces. Filtering and polarizing light often produces contrast that can highlight the flaw. Low incident angles of illumination can create beneficial shadows and highlight crack patterns and mirrors. The purpose of this examination is to locate the flaw origin and, if possible, to characterize the flaw. This usually entails finding a fracture mirror and observing the defect approximately centered therein. Fracture mirrors, as shown in Figures 2 and 3, are telltale indicators of the origin of failure and are readily observable in glasses and fine-grained, dense polycrystalline ceramics. They are less obvious and more vague in coarse-grained, porous ceramics, but still can be used to locate a failure origin.

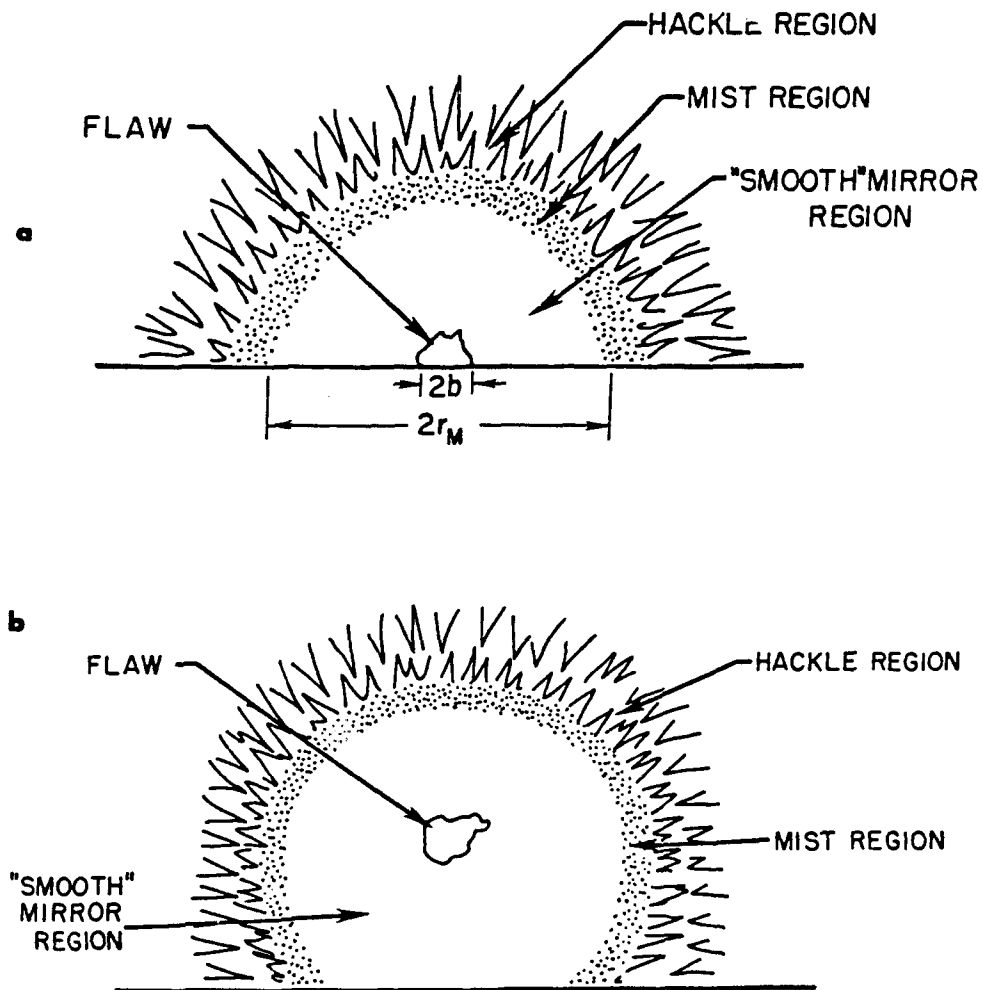
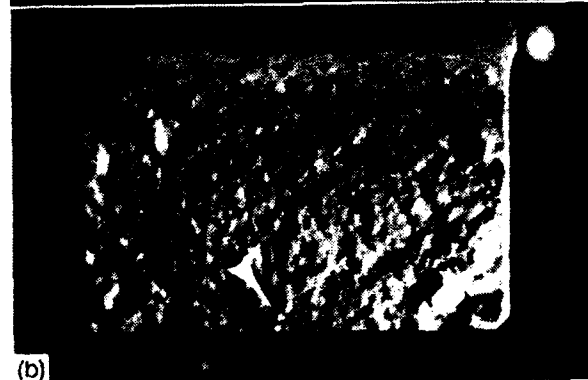
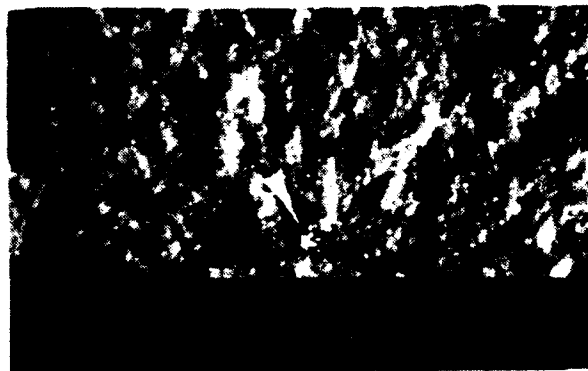


Figure 2. Schematic of the fracture surface of an advanced ceramic which failed in a brittle manner showing (a) a defect at the surface and (b) in the volume and the intrinsic fracture markings. In ceramic terminology "smooth" is a relative term and the fracture mirror can be centered on the flaw or a portion of the flaw.



(a)



(b)

Figure 3. Optical photographs of fracture surfaces from two strong silicon nitrides: (a) was taken on a camera stand and shows a pair of flexure fracture surfaces mounted back-to-back. This dense, fine-grained, hot-pressed specimen has a fairly well defined fracture mirror (arrows), and (b) was taken from a monitor-TV camera viewed on a binocular microscope and shows a sintered silicon nitride flexure specimen. The mirror is not well defined due to subtle microstructural variations and residual porosity. The bottom photograph shows the whole fracture surface; the top is a close-up of the inclusion.

The specimens should be mounted in a holder. A simple alligator clip attached to a stand and having a compliant coating or sheath covering the teeth provides a sturdy and flexible grip. Clays or waxes **should not** be used for mounting because these materials can contaminate the fracture surface and are very difficult to remove. Both halves of the primary fracture surface should be examined, as illustrated in Figures 4 and 5. In the case of flexure specimens, mounting the mating halves back-to-back (tensile surface-to-tensile surface, as shown in Figure 5) can expedite and improve the quality of the analyses. Exterior surfaces should be examined to determine if there is handling or machining damage on the surface which may have initiated the failure.

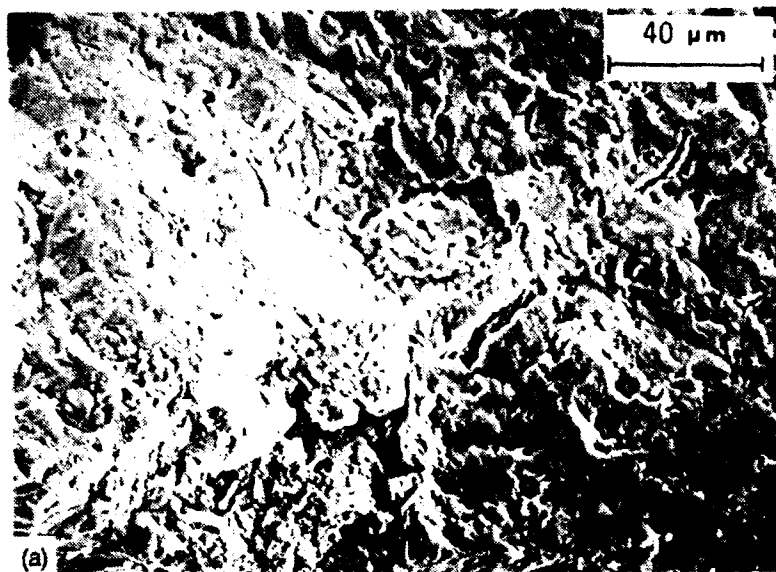


Figure 4a. Failure origin in a sintered alpha silicon carbide flexure specimen:  
(a) is a SEM photograph which suggests the origin is porosity.

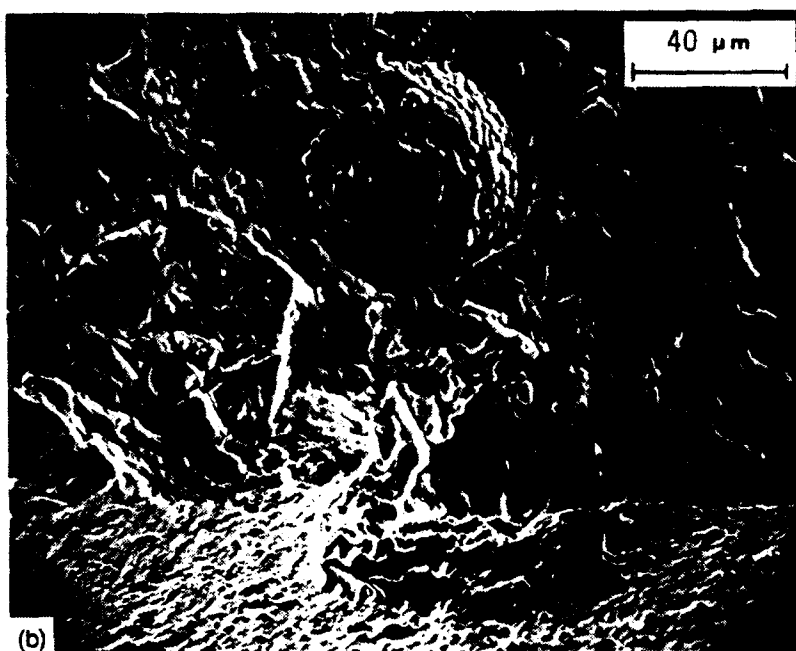


Figure 4b. Clearly reveals a spherical agglomerate  
on the matching fracture surface.

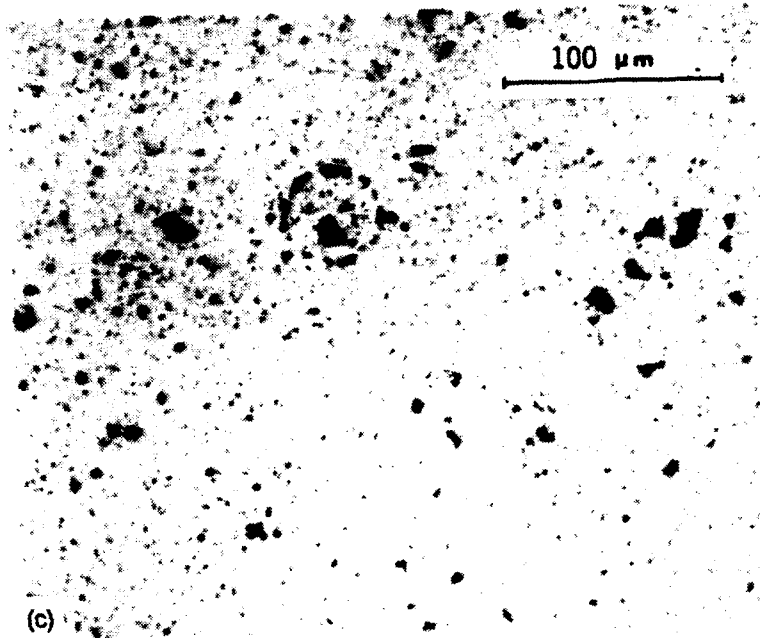


Figure 4c. Is a polished section revealing such defects are common and are much larger than the usual residual microporosity.

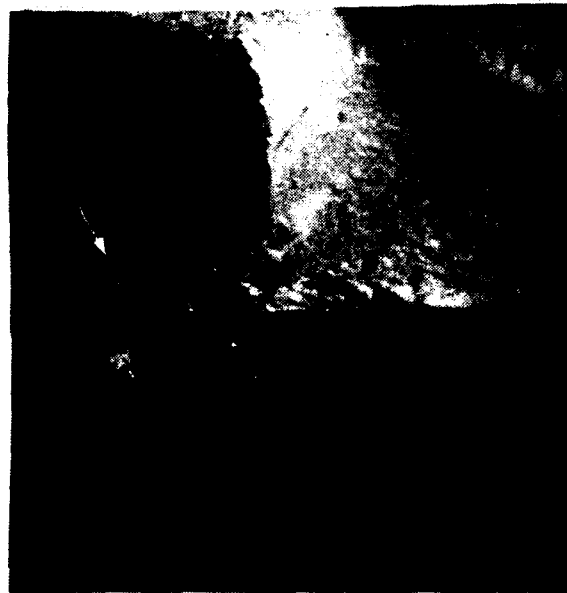


Figure 5. Optical photograph showing matching halves of a reaction-bonded silicon nitride (RBSN) flexure specimen ( $\sigma = 195$  MPa). The defect (arrow) appears to be a pore in one half but is an agglomerate in the other half. The fracture mirror is vague due to the low strength and residual porosity.

All fracture surfaces that are potentially primary must be examined. Multiple fractures are common to high strength, high elastic modulus ceramics that store large amounts of elastic energy during testing. Upon failure, this energy is released and reflects from free surfaces back through the body of the material causing additional failures. Many of the secondary fractures can be eliminated quickly.

It is sometimes necessary, especially with a new, unfamiliar material, to reexamine all of the specimens after the initial examination since a particular flaw is sometimes overlooked or misidentified during the first viewing.

If characterization of the defect is not possible during this step because of the flaw type or size, the optical examination becomes a tool to minimize the time spent during the subsequent examination using a SEM.

Figure 6 shows a camera mounted directly to the binocular microscope which is a great time saver. With built-in zoom ranges of 5 to 1 and beam splitters, it is possible to quickly and efficiently frame, focus, and shoot. Modern built-in video cameras with monitors can be coupled to color printers which give photo-size hard copies in less than one minute without the need to deal with film and negatives. These video images can also be stored in a digital format; i.e., floppy disk. Such optical images can then be retrieved and displayed on a TV monitor or on a SEM monitor. This is a very efficient means of coupling the two methods, and enhanced productivity will result.

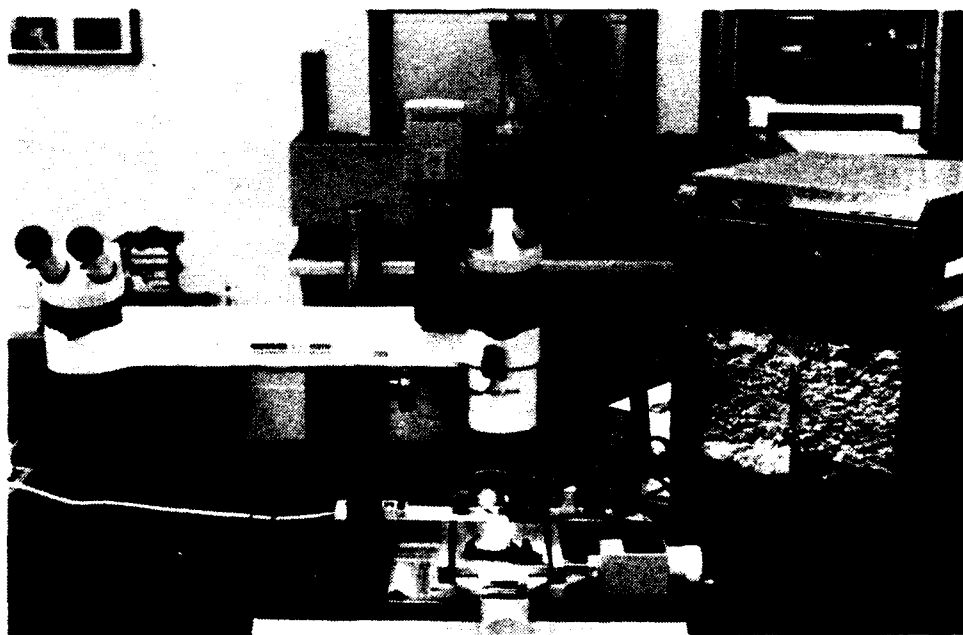


Figure 6. A modern discussion stereomicroscope with simultaneous viewing in both stations and the camera. The color camera sends a signal to a monitor and a printer. Figure 3b was taken with this system. An x-y traversing stage permits measurement of a feature to within  $1\text{ }\mu\text{m}$ .



The standard will recommend that a photograph showing the entire specimen be taken (to show overall crack patterns). It will also strongly recommend a photograph of one or both primary fracture surfaces be taken. Such photographs are very valuable in cueing subsequent electron microscopy, since such images are rather different than optical images, and a reorientation time is often necessary (5 seconds to 1 minute) on the SEM.

### Scanning Electron Microscopy (10X to 2000X)

Optical microscopy is not always adequate to characterize flaws. This is especially true for strong materials which have very small mirror regions and subsequently smaller flaws. Once optical fractography is complete and the flaws characterized as well as possible, a subset of specimens should be prepared for analysis with a SEM. Determination of the number of specimens, and which specimens will comprise the subset, will depend on the intent of the analysis (see Table 2). Analysis for quality control may focus only on specimens which failed below a specific strength, while creation of a database or determination of strength-flaw relationships may require that all specimens be analyzed.

The standard will specify several preparation and cleaning schemes and will include an appendix showing how six common contaminants appear when viewed with the SEM.

The examination's goal is to find and characterize the fracture origin by using the primary fracture mirror with reference to hackle lines.

It may be necessary to acquire an energy or wavelength dispersive X-ray analysis of both the flaw and background in order to determine if there are any chemical differences. In some cases, such as when handling or machining damage are suspected, it may be necessary to tilt the specimen slightly in order to view a portion of the external surfaces. Once the flaw is located, a photograph should be taken at a magnification that provides the maximum amount of information on the flaw and the failure mechanism. This will typically be in the 200X to 1000X range. In many cases, photographs at varying magnifications are necessary to yield all the required information regarding the failure of the specimen.

The most common electron imaging techniques are secondary electron and backscattered electron modes. The secondary electron mode has better spatial resolution, but the atomic-number contrast contribution to the backscattering mode can be exceptionally helpful in discerning inclusions or compositional variation in multiphase materials.

Semiquantitative microchemical analyses can be performed with either energy or wavelength dispersive X-ray analyzers. The former cannot, in most cases, detect elements whose atomic number is less than sodium ( $Z = 11$ ). The latter can detect down to boron ( $Z = 5$ ), but an extremely flat and smooth surface is needed. X-ray maps can show spatial variations of an element, but only to a resolution of 1 micron.<sup>8</sup>

7. HEALEY, J. T., and MECHOLSKY, J. J., JR. *Scanning Electron Microscopy Technique and Their Application to Failure Analysis of Brittle Materials* in Fractography of Ceramics and Metal Failures. J. J. Mecholsky, Jr. and S. R. Powell, Jr., eds., ASTM STP 827, Philadelphia, PA, 1984, p. 157-181.

8. PANTANO, C. G., and KELSO, J. F. *Chemical Analysis of Fracture Surfaces* in Fractography of Ceramic and Metal Failures. J. J. Mecholsky, Jr. and S. R. Powell, Jr., eds., ASTM STP 827, Philadelphia, PA, 1984, p. 139-156.

## Recordkeeping

Recordkeeping during optical microscopy can be as simple as handwritten notes or sketches, or as sophisticated as digitally stored images. Photomacrography (photography through a magnifying camera stand) is inexpensive and practical to 30X but is being supplanted by photomicrography (pictures taken through a microscope). Hyzer<sup>9</sup> discusses the merits of each method.

Optimum recordkeeping shall include at least three photographs taken of each fracture. One set per pair of fracture halves is adequate. These shall include, but not be limited to:

- The entire fracture surface
- The fracture mirror and some surrounding detail
- The defect

## FLAW CHARACTERIZATION

### General

Once a flaw has been found, it remains necessary to characterize it. Hopefully, this can be done unequivocally. The defect may be intrinsic to the manufacture of the material, such as agglomerates, large grains or pores or it may be extrinsic such as handling damage, pits from oxidation or corrosion, or cracks nucleated from cavitation at high temperature. Machining damage could be considered intrinsic to the extent that the damage is a natural consequence to a specific machining fabrication procedure, but we have chosen to regard it as extrinsic, since machining damage usually occurs after material manufacture and is very specific to the precise process. It is beyond the scope of this report to discuss the origin of the defects (the Bibliography Section contains references which comprehensively cover the topic).

The standard will require that defects be characterized by two mandatory and one optional aspects, as shown in Table 4.

Table 4. FLAW CHARACTERIZATION

Identity	Location	Size
Nomenclature: Volume or surface distributed	Volume (bulk), Surface, or Edge	Optional, only as a measure of scale

9. HYZER, W. G. *Photography, Macro or Micro* in Research and Development, v. 26, no. 6, June 1975, p. 22-25.

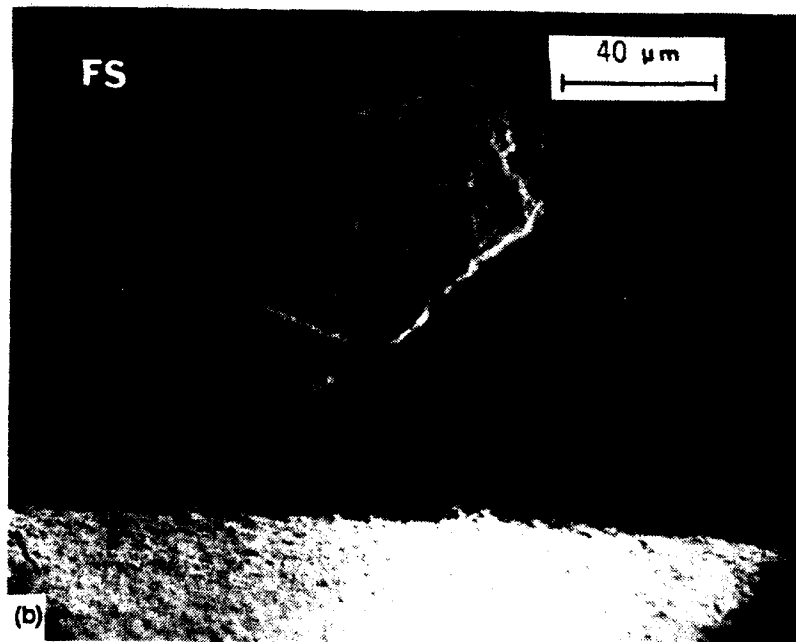


Figure 7. Failure origin in a RBSN flexure specimen broken at room temperature ( $\sigma = 316$  MPa): (a) is an optical photograph showing the defect is a white spot, and (b) is a SEM photograph showing the defect is a simple pore. "T" denotes the tensile surface of the bend bar; "FS" denotes the fracture surface. Tilting a specimen back a bit helps detect machining damage or nearby surface-connected features, although foreshortening of the flaw will occur.

## Identity

The defect identity will be characterized wherever possible by a phenomenological approach which states what the defect is, and not how it appears under a particular mode of viewing. Figure 7 shows a pore in a reaction-bonded silicon nitride (RBSN) which appears as a white spot when viewed optically, but the SEM image clearly identifies the defect as a simple pore. Similar defects were reported by Pasto et al.<sup>10</sup> and Richerson et al.<sup>11</sup>

Appendix 2 lists a suggested nomenclature to specify defect identity and Appendix 3 provides examples of each defect type. It is an expansion to the list given earlier<sup>1</sup> and is generally consistent with the nomenclature used by the experts cited in the bibliography. We have delineated the defects into two categories (surface or volume defects) with regard to their inherent spatial distribution in a ceramic. Machining damage is intrinsically a surface defect. Inclusions are usually volume distributed. The standard practice will include an expanded version of the catalog as an Appendix.

Porosity can manifest itself in a variety of forms<sup>1,2,12,13</sup> including discrete large voids or zones of locally higher porosity, as shown in Figure 8. This can lead to drastically different measured strengths for specimens taken from nominally identical components or plates.<sup>1,2</sup> Therefore, it was decided to include three possible porosity defect categories, as given in Appendix 2 and illustrated in Figure 9. We recognize, however, that a particular defect may have attributes of several types. For example, porous regions often have a small pore associated with them.

The defects in many advanced ceramics are so small ( $< 50 \mu\text{m}$ ) that accurate characterization can only be made via SEM. Preliminary assessments can be made with optical microscopy but, as was shown in Figure 7, what may optically seem to be an agglomerate or inclusion could very well be a pore. Nevertheless, optical microscopy is an essential adjunct since certain features such as a telltale color or reflectivity are completely lost in electron microscopy.

It should also be noted that sometimes the true flaw may be incompletely exposed in a fracture mirror. This often occurs in instances where a defect is oriented at an angle to the fracture surface and only a portion of its true size is exposed.<sup>14</sup> Another possibility is illustrated in Figure 10 where a pore was detected at a failure origin, but only when transmitted light was used was it recognized that the pore was part of a band of porosity.

10. PASTO, A. E., NEIL, J. T., and QUACKENBUSH, C. L. *Microstructural Effects Influencing Strength of Sintered Silicon Nitride* in *Ultrastructure Processing of Ceramics, Glasses and Composites*. L. Hench, and D. Ulrich, eds., John Wiley and Sons, New York, 1984, p. 476-489.
11. RICHIERSON, D. W., SMYTH, J. R., and STYHR, K. H. *Material Improvement Through Iterative Process Development*. Ceram. Eng. Sci. Proc., v. 4, no. 9-10, 1983, p. 841-852.
12. RICE, R. W. *Processing Induced Sources of Mechanical Failure Ceramics* in *Processing of Crystalline Ceramics*, H. Palmour, R. Davis, and T. Hare, eds., Plenum Press, New York, 1978, p. 303-319.
13. RICE, R. W. *Pores as Fracture Origins in Ceramics*. J. Mat. Sci., v. 19, 1984, p. 895-914.
14. KIRCHNER, H., GRUVER, R., and SOTTER, W. *Characteristics of Flaws at Fracture Origins and Fracture Stress - Flaw Size Relations in Various Ceramics*. Mat. Sci. Eng., v. 22, 1976, p. 147-156.

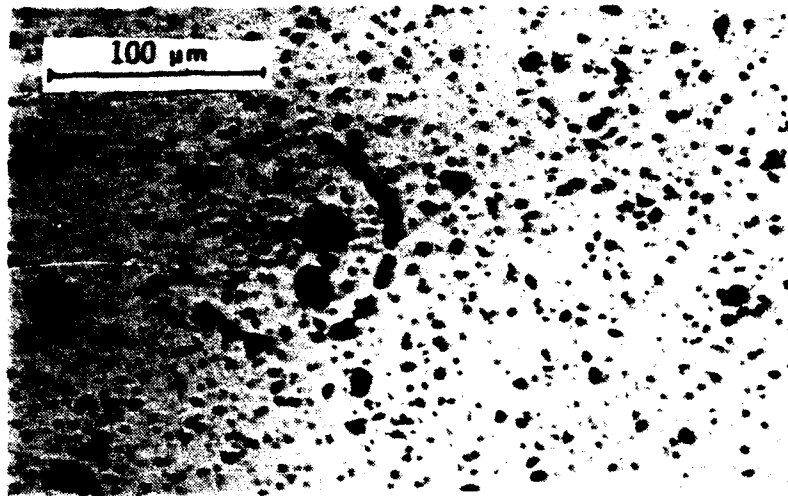


Figure 8. An optical micrograph of a polished section of a RBSN revealing two different types of gross porosity which are potential strength-limiting flaws, and the much smaller normal microporosity.

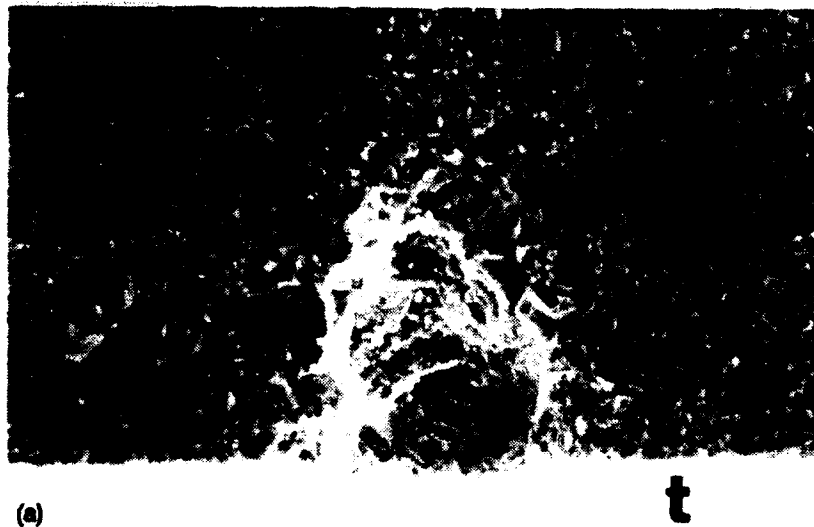


Figure 9. Scanning electron micrographs of porosity-related defects, all are the same grade of 99.9% sintered alumina<sup>2</sup>; (a) shows a discrete pore.

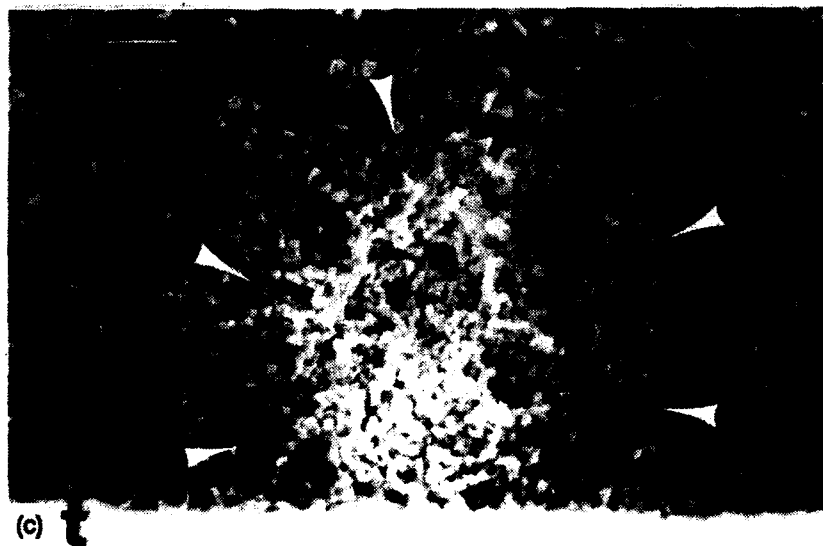


Figure 9 (cont'd). Scanning electron micrographs of porosity-related defects, all are the same grade of 99.9% sintered alumina<sup>2</sup>; (b) shows a discrete pore; (c) shows a porous region.

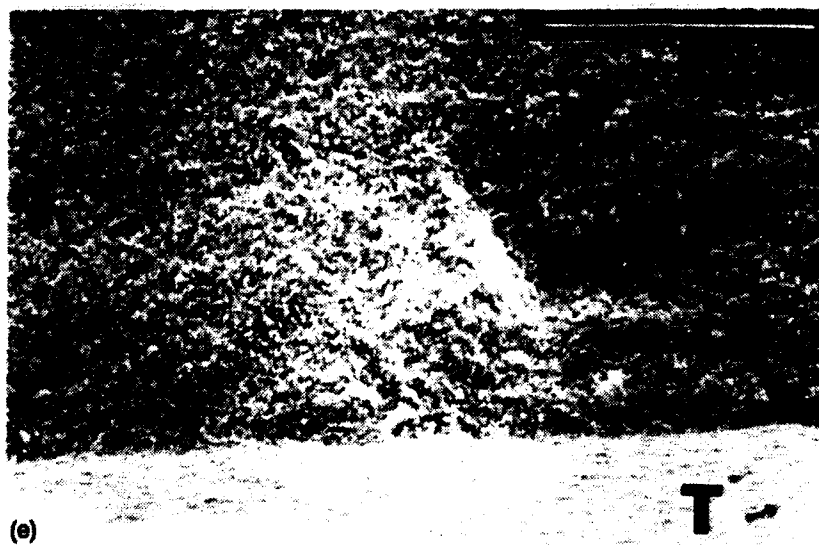
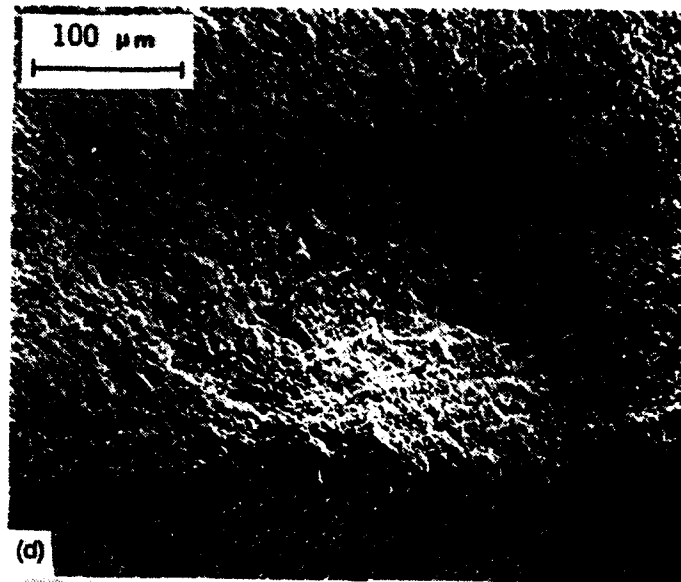


Figure 9 (cont'd). Scanning electron micrographs of porosity-related defects, all are the same grade of 99.9% sintered alumina<sup>2</sup>; (d and e) show planar porous seams.

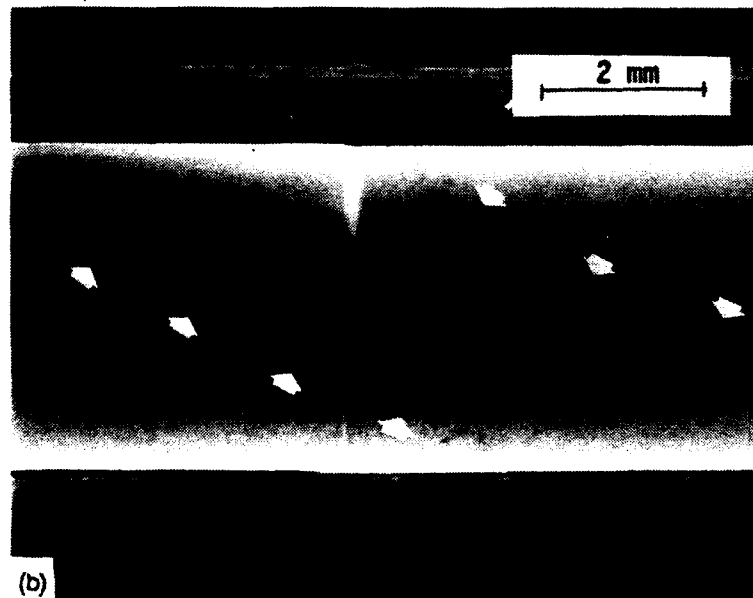
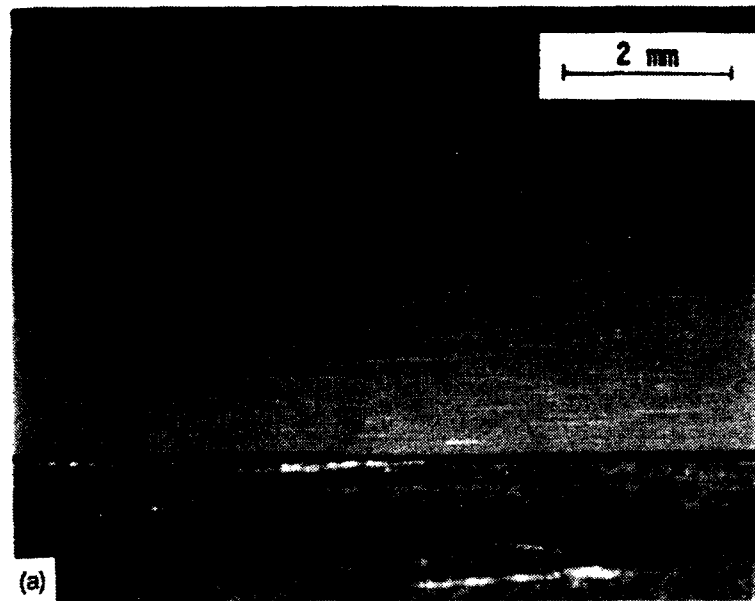


Figure 10. Optical photographs of the tensile surface of a 99.9% alumina flexure specimen ( $1000^{\circ}\text{C}$ ,  $\sigma = 275 \text{ MPa}$ ); (a) shows the surface with the lighting at an angle which tends to highlight the surface machining striations. The fracture surface is in the middle; (b) shows the identical specimen but with the light shining through the sides of this translucent material. The failure origin is marked by the black arrow, but it is associated with a band of porosity (white arrows).



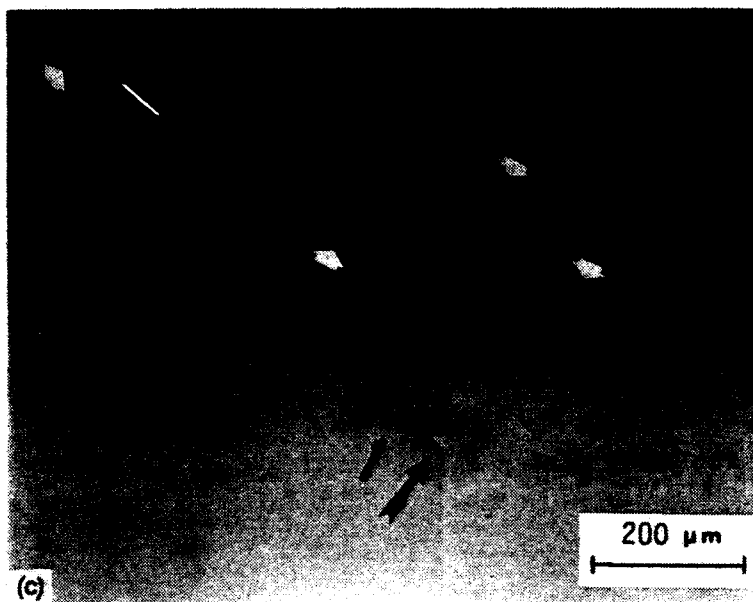


Figure 10 (cont'd). Optical photographs of the tensile surface of a 99.9% alumina flexure specimen ( $1000^{\circ}\text{C}$ ,  $\sigma = 275 \text{ MPa}$ ); (c) shows a close-up showing the fracture origin.

### Location

After a flaw is identified, the next characterization step is to specify its location. The standard will not prescribe that this be measured precisely, but only requires that the location be specified as to whether it is in the volume (bulk), at the surface, or at an edge. The location of defects is of critical importance to designers, since surface or edge defects may behave very differently than identical flaws located in the bulk. Fracture mechanics considerations show that a defect located at or near a surface will have a higher stress intensity than if in the bulk at the same stress.

Many ceramics are sensitive to time-dependent, environmental effects, and surface-connected flaws may be preferential failure origins.<sup>15,16</sup> Intrinsically **volume** distributed defects, such as pores or agglomerates in a ceramic plate or component, may be **located** at the volume, surface or edge of a test specimen, simply due to the sampling inherent to preparing the test specimen. On the other hand, intrinsic **surface** defects can **only** exist at the surface or at an edge.

15. QUINN, G. D., and KATZ, R. N. *Time Dependent High Temperature Strength of Sintered  $\alpha$ -SiC*. J. Am. Ceram. Soc., v. 63, no. 1-2, 1980, p. 117-119.

16. QUINN, G. D. *Review of Static Fatigue in Silicon Nitride and Silicon Carbide*. Cer. Eng. and Sci. Proc., v. 3, no. 1-2, 1982, p. 77-98.

The assessment of flaw location must be done carefully if the defects are near the surface, especially in flexure specimens. What may optically appear to be a surface flaw can be shown in the SEM to be a volume defect located near a surface, as shown in Figure 11. Figure 12 shows volume defects which are located at or near a flexure specimen edge. Figure 13 shows genuine machining damage at a chamfer.

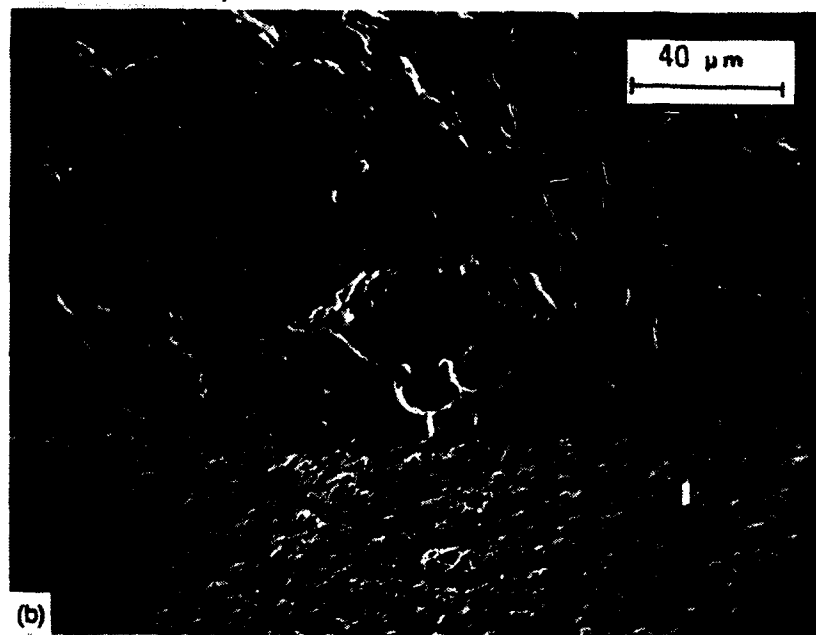
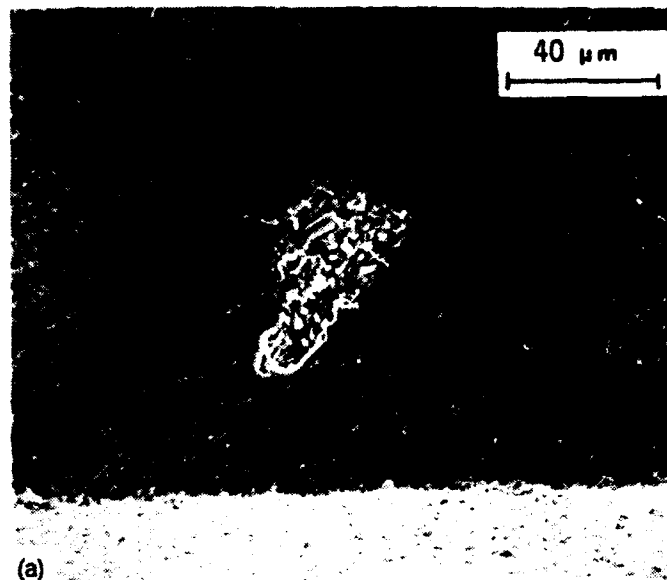


Figure 11. Intrinsically **volume** distributed pores in flexure specimens: (a) is located in the bulk of sintered beta silicon nitride ( $\sigma = 460$  MPa), (b through d) are sintered alpha silicon carbide. (b) is a pore near the surface in a flexure specimen ( $\sigma = 408$  MPa).

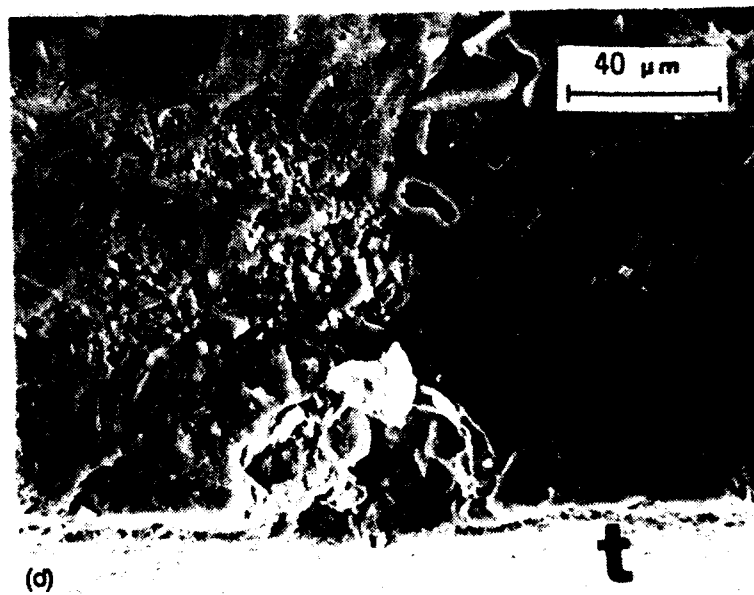
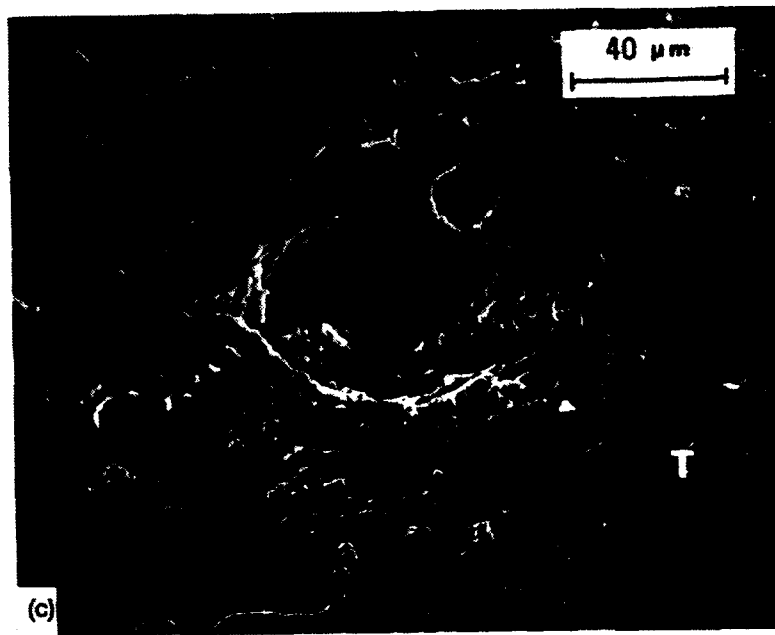
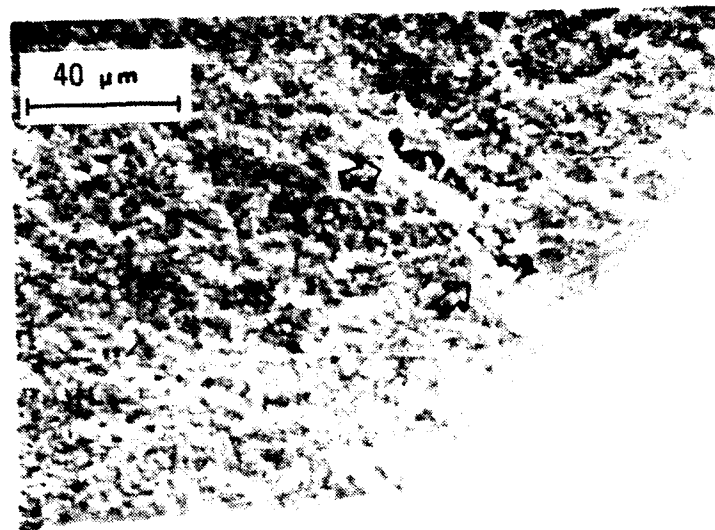


Figure 11 (cont'd). Intrinsically volume distributed pores in flexure specimens: (c and d) are surface-located pores in 1200°C stress rupture specimens (350 MPa at 0.9 hours, and 260 MPa at 315 hours).



(a) **T**

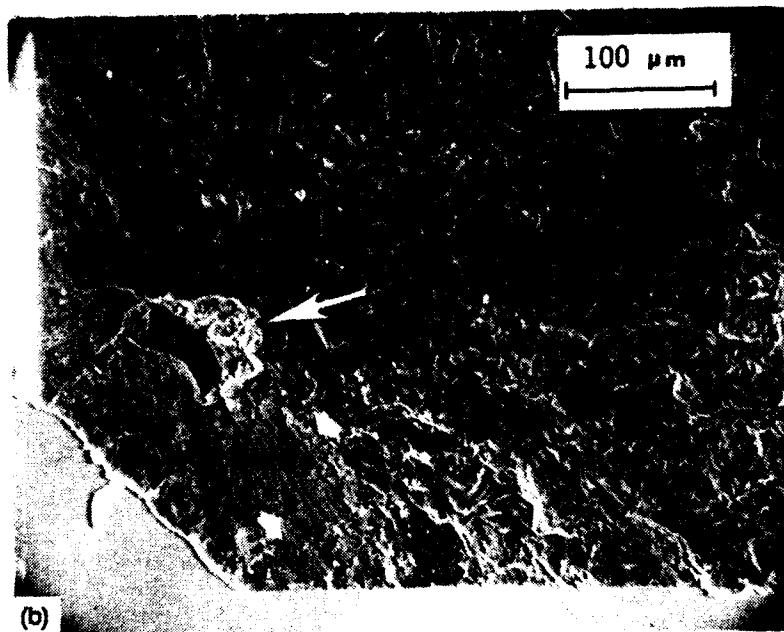


Figure 12. Volume-distributed defects which were located at the edge (chamfer) of flexure specimens; (a) shows a pore in a sintered silicon nitride stress-rupture specimen (1200°C, 550 MPa, 22 hours); (b) shows a pore in sintered alpha silicon carbide with nearby large grain (small arrows) ( $\sigma = 449$  MPa).

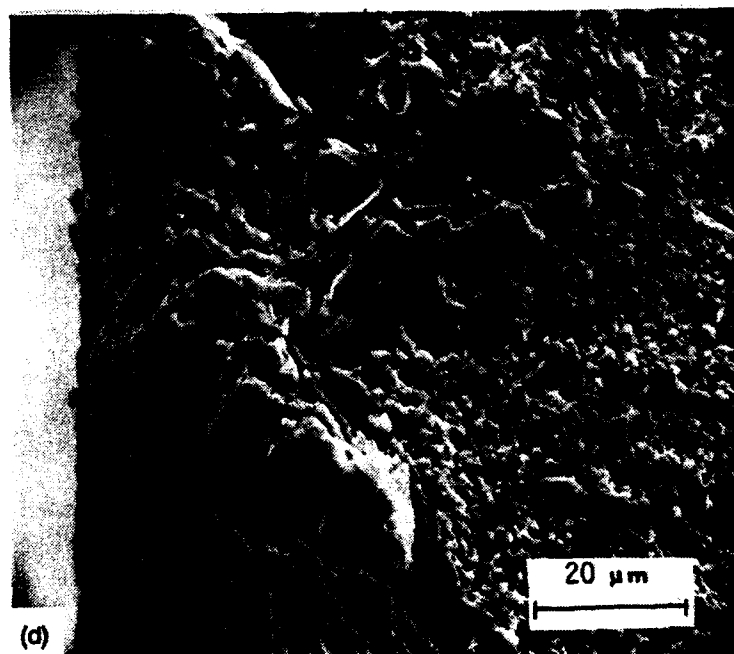
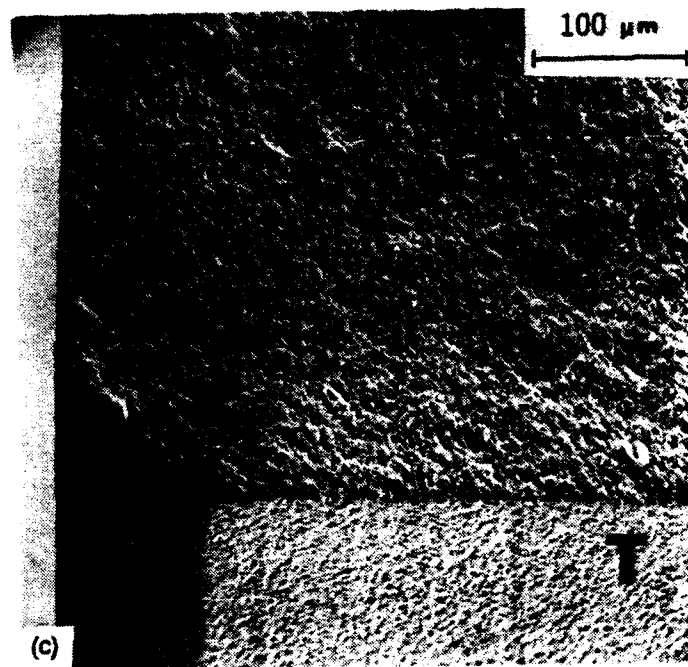


Figure 12 (cont'd). Volume-distributed defects which were located at the edge (chamfer) of flexure specimens; (c and d) Shows a large grain cluster in a sintered 99.9% alumina ( $\sigma = 404$  MPa).

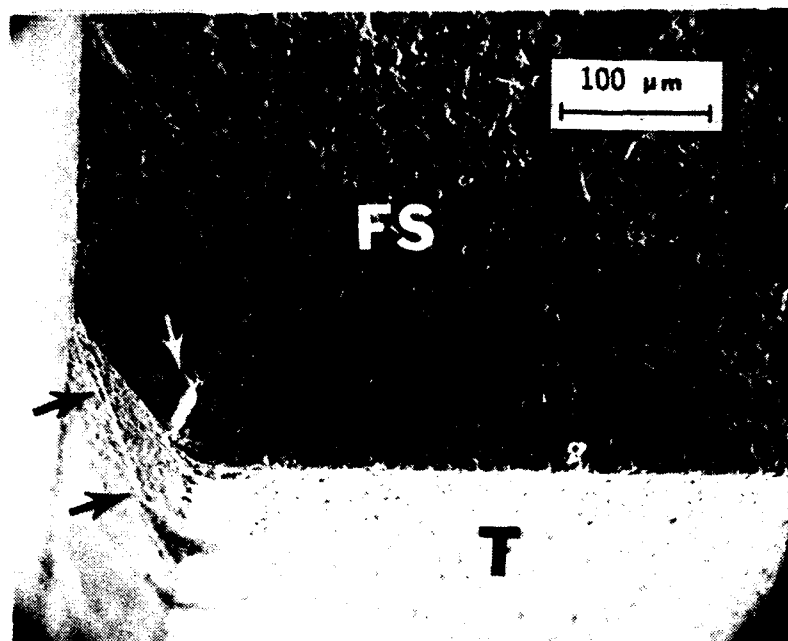


Figure 13. SEM of a RBSN flexure specimen that broke at room temperature ( $\sigma = 264$  MPa). The defect is machining damage at the chamfer. Tilting the specimen back reveals sporadic chips and the long striation (black arrows) at an angle. This attests to the grinding direction not being longitudinal, which caused the excessive machining damage and chipping.

## Size

Flaw size characterization is only required by the standard in a qualitative sense as necessary to generally clarify the nature of the defects (e.g., the  $20\ \mu\text{m}$  pores) or to permit a qualitative comparison to average microstructural features such as the average grain size.

Precise flaw measurements are problematic since: the defect's true size may not necessarily be revealed on the fracture surface; the flaw periphery may be difficult to appraise; and fracture mechanics analysis of most flaws is difficult, if not impossible, due to the shape complexity. Residual stresses, local variations in fracture toughness (due to the flaw interacting with the material),<sup>17</sup> and elastic property mismatches are complicating factors. The literature shows mixed results in attempts to correlate flaw size to strength. The data often gives extreme scatter or no relationship at all.<sup>3,18,19,20</sup> The data are usually interpreted by trends.<sup>21-23</sup> Evans et al.<sup>21</sup> in a widely cited study showed strength versus flaw size on one graph for six

17. BAUMGARTNER, H. R., and RICHESON, D. W. *Inclusion Effects on  $\text{Si}_3\text{N}_4$*  in Fracture Mechanics of Ceramics, Vol. 1. R. Bradt, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1984, p. 367-386.
18. SANDERS, W. A., and BAAKLINI, G. Y. *Correlation of Processing and Sintering Variables with the Strength and Radiography of  $\text{Si}_3\text{N}_4$* . Adv. Cer. Mat., v. 3, no. 1, 1988, p. 88-94.
19. SESHADRI, S. G., and SRINIVASAN, M. *On Estimating Fracture Toughness by Intrinsic Flaw Fractography for Sintered Alpha Silicon Carbide*. Com. Am. Ceram. Soc., April 1981.
20. GEE, M. G., and MORRELL, R. *Fracture Mechanics and Microstructure* in Fracture Mechanics of Ceramics, Vol. 8., R. Bradt, A. Evans, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1986, p. 1-22.
21. EVANS, A. G., MEYER, M. E., FERTIG, K. W., DAVIS, B. I., and BAUMGARTNER, H. R. *Probabilistic Models of Defect Initiated Fracture in Ceramics*. J. Non-Destruct. Eval., v. 1, 1980, p. 111-114.
22. RICE, R. W. *Fractographic Determination of  $K_{Ic}$  and Effects of Microstructural Stresses on Ceramics*. Fractography of Glasses and Ceramics II. To be published.
23. EVANS, A. G., and TAPPIN, G. *Effects of Microstructure on the Stress to Propagate Inherent Flaws*. Proc. Br. Cer. Soc., v. 20, 1972, p. 275-297.

different flaw populations in a hot-pressed silicon nitride with magnesia additive. This study was especially instructive in showing the different severities of flaws in a given material. Surface cracks from machining damage were the most severe and could be reasonably modelled by simple fracture mechanics analysis. Rice<sup>22</sup> has strongly advocated quantitative analysis of flaw size, and also reached the conclusion that the defects which are most amenable to analysis are machining defects. However, residual stresses are often present. In summary, quantitative measurements of flaw size can at times be very valuable, but this is a difficult, often subjective, analysis and it is not required by the standard practice.

We realize that defect characterization will sometimes be equivocal. Fracture mirrors may be so vague as to be undiscernible. The defects themselves may be difficult to find or specify. Defects sometimes exist together (e.g., an agglomerate and a surrounding pore, or a pore with large grain), in which case some judgement is required as to which is more fundamental or dominant. An example of this exercise in judgement is in Figure 12b which shows a large grain adjacent to a pore. The large grain shows hackle marks which clearly identify that the pore was the dominant defect. There is no reason why defects cannot be described by paired expressions (e.g., a pore/large grain). Figure 14 is another example of an equivocal characterization.

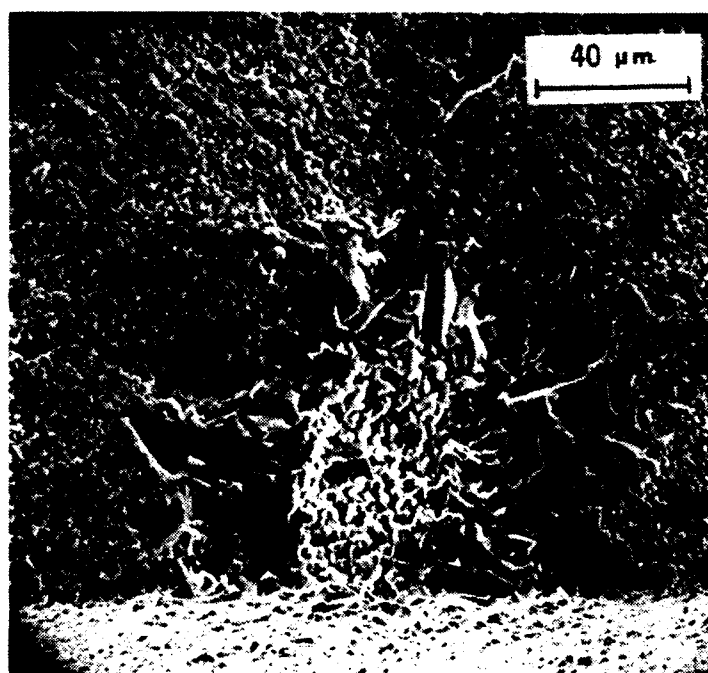


Figure 14. Strength-limiting defect in a 99.9% sintered alumina flexure specimen ( $\sigma = 396$  MPa). This defect could alternately be identified as a pore or a cluster of large grains.

Defect interactions are a potentially serious characterization and design problem. Figure 15 shows a semi-elliptical, machining damage crack that has interacted with a volume-distributed pore that is located at the surface. Evans and Tappin<sup>23</sup> discussed flaw interactions and linking prior to catastrophic breakage. They further pointed out that a flaw located near a surface will create a stress concentration in the ligament between the defect and the surface. The ligament will be apt to break prior to catastrophic failure and will produce a hybrid defect.

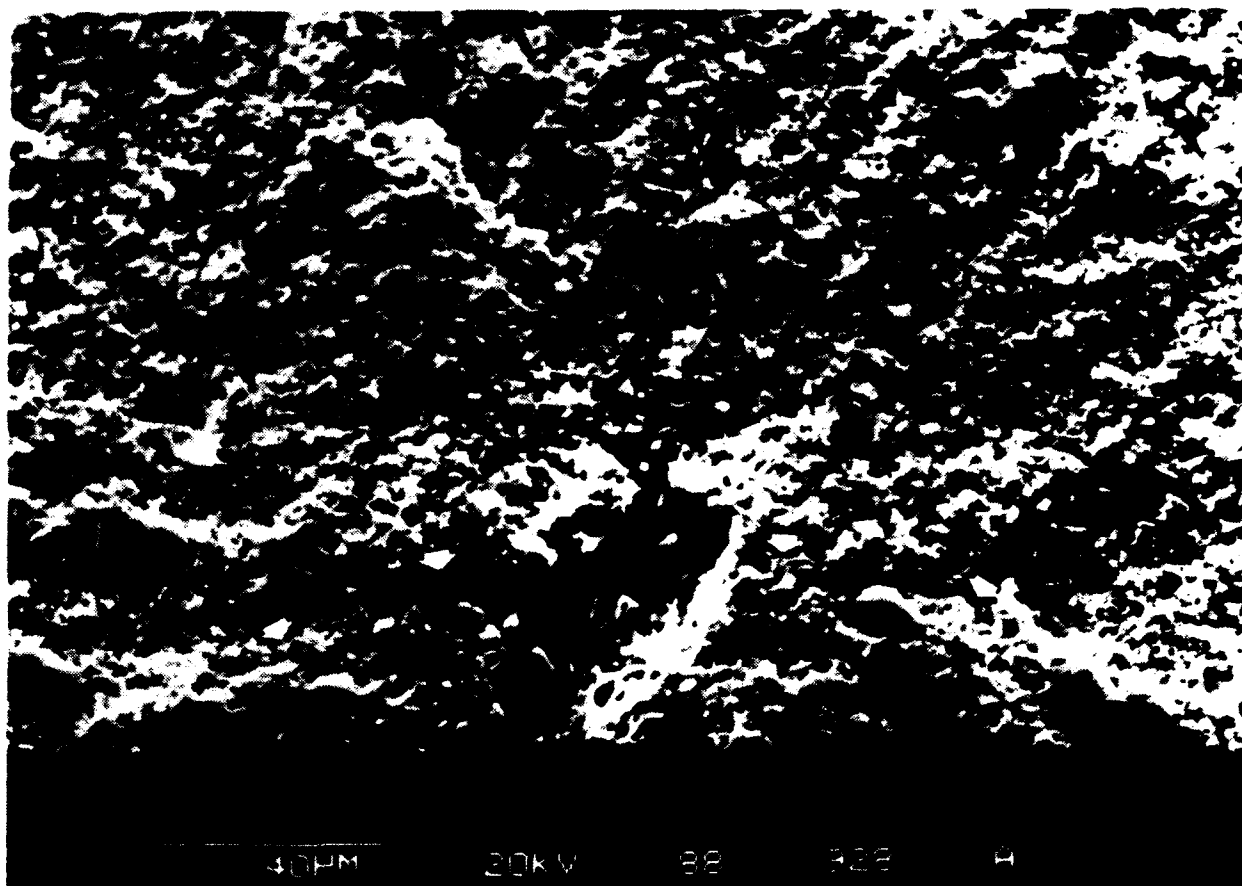


Figure 15. Interactive semi-elliptical machining damage (arrows) and a large pore in a sintered silicon nitride. A few clay particles contaminate the fracture surface ( $\sigma = 601$  MPa).

The flaw definitions in the standard are primarily in the context of there being gross aberrations in the material microstructure; as such, they often stand out readily against the background of the normal microstructure. As fabricators improve materials by careful process control, and as they manage and eliminate these defects, ceramics will become strength-limited by "defects" that are within the normal range of sizes of microstructural features. The "defects" will typically be commensurate with the average grain size and will presumably represent the large-sized end of the size distribution of the microstructural features (Petch graphs of strength versus average grain size may become fashionable again!). The term "defect" has to be used rather loosely in these cases. Rice<sup>24</sup> has considered these origins-of-failure and refers to them as "mainstream microstructural features" or "basic microstructural defects." He terms them as intrinsic defects, whereas the abnormal microstructural features, such as large pores or grains, he calls extrinsic. We prefer to term the latter as intrinsic as well, since we take a manufacturer's perspective that such defects are present as a consequence of the

24. RICE, R. W. *Failure Initiation in Ceramics, Challenges of NDE and Processing in Ceramic Developments*. C. Sorrel, and B. Ben-Nissim, eds., Materials Science Forum, Vol. 34-36. Trans. Tech. Bbl. Ltd., Switzerland, 1988, p. 1057-1064.



manufacturing process. Recent studies on well processed, high-strength silicon nitrides,<sup>25,26</sup> and aluminum oxynitrides<sup>27</sup> have shown the origin-of-failure can reach the size of average microstructural features. In these cases, the origin will blend well into the background microstructure and will be extremely difficult to discern even with careful SEM scrutiny.

The nomenclature of Appendix 2 shall suffice for this class of failure origins, but it is very important that the user distinguish whether the "defect" is an abnormally large feature or is a "mainstream microstructural feature." Therefore, the standard shall require that, in conjunction with the fractographic analysis, a representative metallographic polished section be prepared as well so that the average microstructure can be evaluated.

Another problem arises if a "defect" is not a discrete feature but is a region in the material where the microstructure deviates a minor amount from the norm. Brook<sup>28</sup> illustrates this for an alumina where inhomogeneous sintering led to variations of microporosity which acted as defects. These could be labelled as porous seams or zones, but they could be very difficult to detect if the material fractures intergranularly.

## REPORTING RESULTS

Once the fractographic analysis is complete, the results must be stored and reported. Fractographic results are typically recorded on photographs, and it is worthwhile to organize these in a systematic fashion to aid retrieval and analysis. The standard will recommend that an optimum sequence is to retain three photographs for each fracture surface; the whole fracture surface, the fracture mirror with some hackle, and a closeup of the defect. Of course, this is an ideal and it is recognized that it may not always be possible to allocate the time and resources to it.

The standard will recommend several schemes to present the complementary fractographic and mechanical testing information. Labelled Weibull graphs, as shown in Figure 16, are an excellent scheme to do such, and at a glance permit an assessment of the applicability of the Weibull analysis. Multiple flaw populations can be directly related to irregularities on the strength curve.

Fractographic montages, such as depicted in Figure 17, permit photographs to be arranged on a single worksheet around a graph of the mechanical test results. A mass of optical and SEM photos can be organized in this fashion and notes and observations scrawled on such a worksheet. Patterns can emerge, such as the relationship between the large-grain failure origins and the low-strength end of the Weibull graph. The montages are relatively easy to store in map or blueprint cabinets, and the ease of retrieval encourages fractographic reassessment or comparisons of one worksheet to another. (The preparation on this paper involved examining thousands of fractographs accumulated over the course of 17 years, a task made much easier by their organization on montages.) This is far better than dumping unlabelled photographs into the back of a file cabinet or pasting two or three photographs per page into a laboratory notebook. Poster board material is recommended over paper sheets, which are liable to tear or become flimsy with repeated usage.

25. LIU, K. D., and BRINKMAN, C. R. *High Temperature Tensile and Fatigue Strengths of Silicon Nitride* in Proceedings of the 27th Automotive Technology Development Contractors Coordination Meeting, SAE, Warrendale, PA, 1989, p. 235-244.

26. CARRUTHERS, D. Private Communication, 1989.

27. QUINN, G. D., CORBIN, N. D., and MCCAULEY, J. W. *Thermomechanical Properties of Aluminum Oxynitride Spinel*. Am. Cer. Bull. v. 63, no. 5, 1984, p. 723-729.

28. BROOK, R. J. *Microstructural Design for Engine Components* in Ceramic Materials and Components for Engines. W. Bunk, and H. Hausner, eds., DKG, Berlin, 1986, p. 477-484.

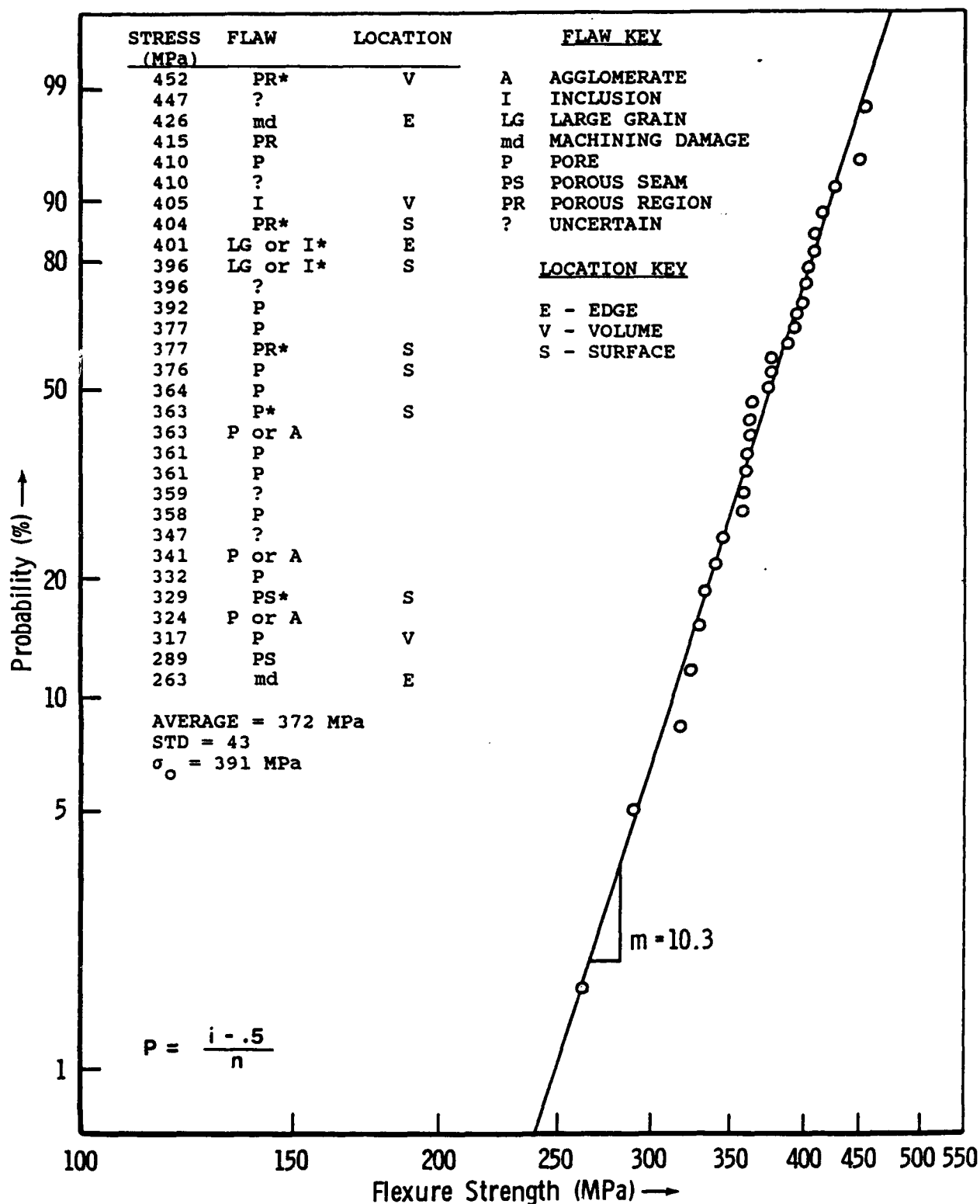


Figure 16. A labelled Weibull graph which incorporates the strength and fractographic results. Four-point flexure strengths at room temperature in a sintered 99.9% alumina.<sup>2</sup> All specimens were optically examined. Specimens with an asterisk were SEM examined.

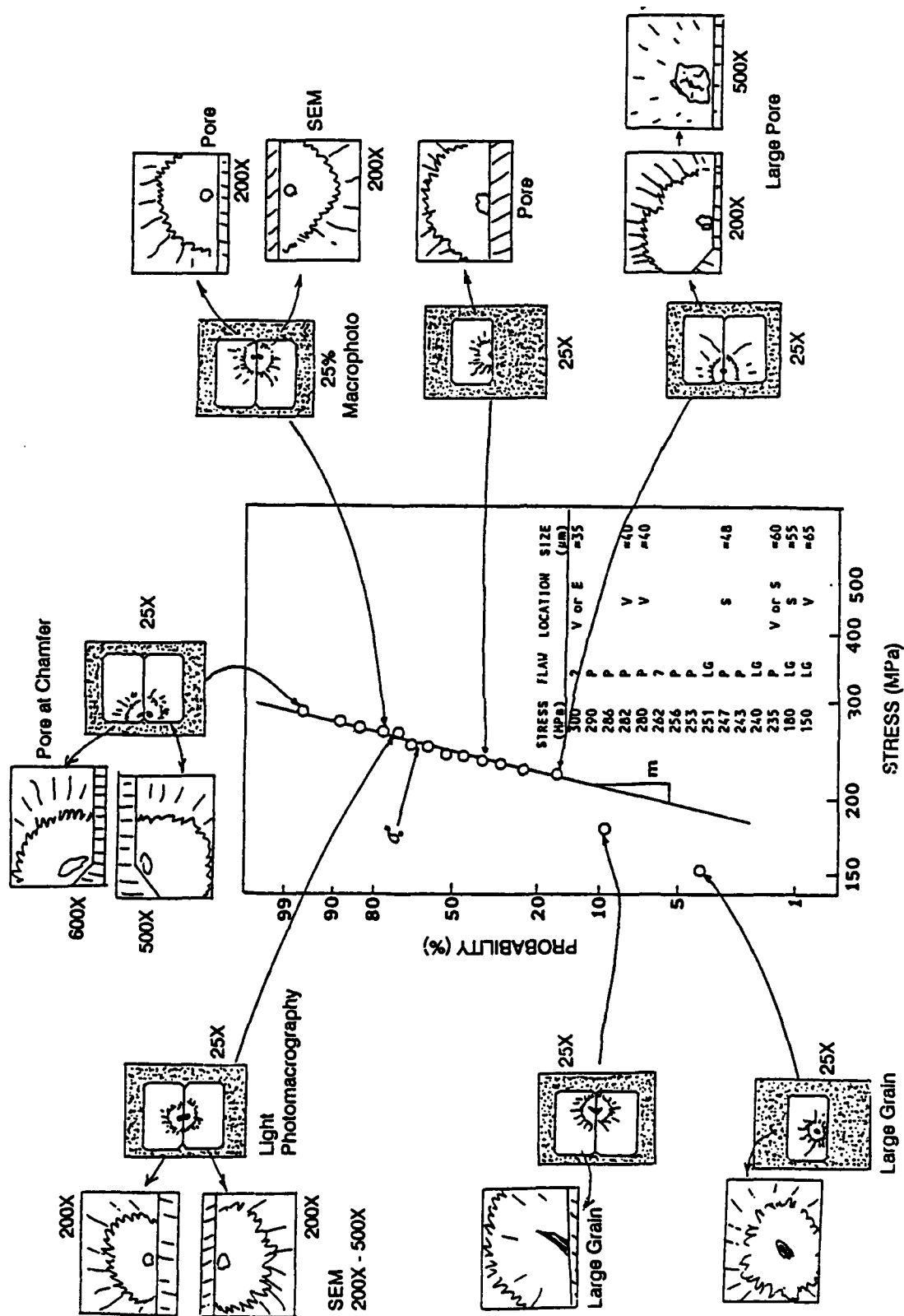


Figure 17. A schematic of a fractographic montage which combines a labelled Weibull graph and fractographic photographs and notes in a fashion which emphasizes the complementary value of fractography and mechanical test results.

Montages can be carefully and neatly assembled for publication and are finding wide-spread acceptance.<sup>1,25,29,30</sup> Figure 18 shows a fine example for tension-fatigue results in a silicon nitride. Four representative specimens had three photographs each displayed; usually the overall fracture surface, the mirror, and the defect. This is the sequence recommended by the standard and is a very convincing presentation of the fractography which can be readily understood and appreciated by a user.

The detailed fractographic report must include details on the equipment used, the modes of viewing for each specimen, and the criteria used for selecting specimens.

### SUMMARY

A standard practice for fractographic analysis is being prepared as a U. S. Army Military Standard. The primary goal is to encourage fractography as an adjunct to mechanical testing. It is directed at finding and characterizing strength-limiting defects in monolithic or "simple" composite advanced ceramics. The standard will be applicable to quality control, materials development, and design purposes. It specifies visual, optical microscope, and SEM examinations but with flexible procedures and sampling criteria. The standard will present guidelines and recommendations rather than dictated precise procedures.

Flaw characterization will consist of three attributes: type, location, and size. A phenomenological nomenclature has been devised and takes into account whether the defects are intrinsically volume or surface distributed. Flaw location shall be qualitatively specified with respect to its location in the volume (bulk), surface, or edge of a test specimen or component. Flaw size shall be measured only to the extent to approximately characterize the flaw population with respect to the microstructure.

Suggested reporting schemes will be given in the standard. The standard will include six appendices including a bibliography, fracture patterns in common test specimens, common contaminants, a defect nomenclature and definitions, a defect catalog, and fractographic montages. The primary purposes of this paper have been to outline the objectives and form of the prepared standard, and to solicit constructive criticism.

29. KAWAI, M., FUJITA, H., KANKI, Y., ABE, H., and NAKAYAMA, J. *Tensile Testing of Sintered Silicon Carbide and Nitride in Ceramic Materials and Components for Engines*. Proceeding of the Conference at Hakone, Japan, 1983.
30. MUSUDA, M., MATSUI, M. *Fatigue in Ceramics, (Part 4) - Static Fatigue Behavior of Sintered Silicon Nitride Under Tensile Stress*. J. Ceram. Soc. Japan, Int. Ed., v. 98, no. 86, 1990, p. 88-97.

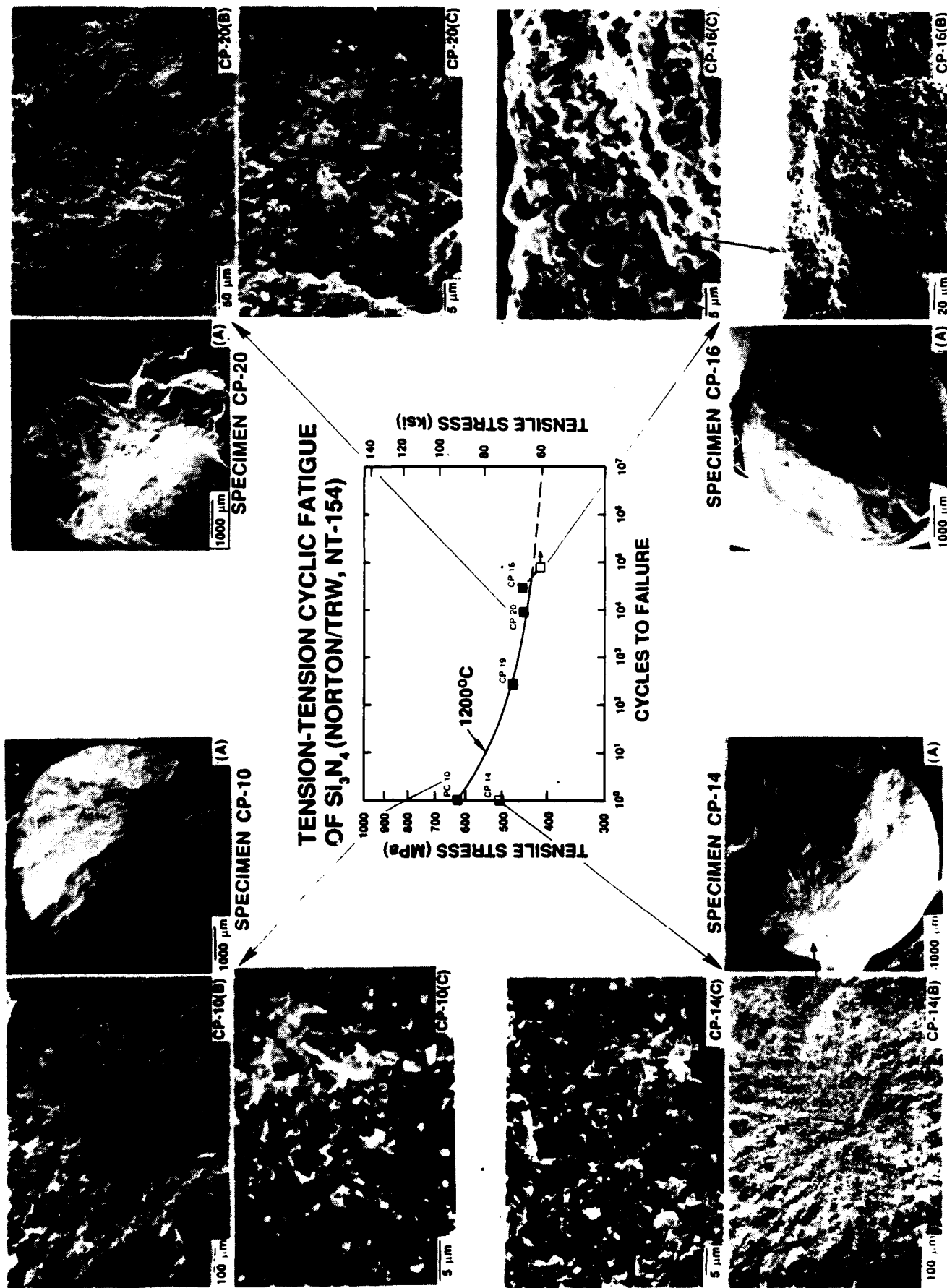


Figure 18. A fractographic montage for mechanical fatigue in tension for a hot-isostatically-pressed silicon nitride.<sup>25</sup>

### **ACKNOWLEDGMENT**

Gail Meyers provided helpful comments regarding electron microscopy and helped prepare the section on such in the draft Military Standard. Arvid Pasto at GTE Laboratory in Waltham, MA provided useful photomicrographs for inclusion in Appendix 3.

Supplementary Note: If you have any questions, comments, or input about this practice please contact the authors at:

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ATTN: SLCMT-EMC  
Arsenal Street  
Watertown, MA 02172-0001

or call Jeffrey Swab (617) 923-5410.

## APPENDIX 1. A SELECT BIBLIOGRAPHY ON FRACTOGRAPHY AND DEFECTS IN CERAMICS

### Books on Advanced Ceramics Fractography

1. Fractography of Glasses and Ceramics. Advances in Ceramics, Vol. 22, J. Varner and V. Frechette, eds., American Ceramic Society, Westerville, Ohio, 1988.
2. Fractography of Ceramic and Metal Failures. J. Mecholsky and S. Powell, Jr., eds., American Society of Testing and Materials, ASTM STP 827, Philadelphia, 1984.
3. Fracture Mechanics of Ceramics, Vol. 1, Concepts, Flaws and Fractography. R. Bradt, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1974.

### Microscopic Techniques

1. HEALEY, J. T., and MECHOLSKY, J. J., Jr. *Scanning Electron Microscopy Techniques and Their Application to Failure Analysis of Brittle Materials* in Fractography of Ceramics and Metal Failures, ASTM STP 827, 1984, p. 157-181.
2. PANTANO, C. G. and KELSO, J. F. *Chemical Analysis of Fracture Surfaces* in ASTM STP 827, 1984, p. 139-156.
3. HYZER, W. G. *Photography, Macro or Micro* in Research and Development, v. 26, no. 6, June 1975, p. 22-25.
4. DAHLBERG, P. *Instrumentation for Surface Analysis* in Research and Development, v. 26, no. 6, June 1975, p. 16-20.
5. FRECHETTE, V. D. *The Fractography of Glass* in Introduction to Glass Science. L. Pye, H. J. Stevens, and W. C. LaCourse, eds., Plenum Press, New York, 1972, p. 433-450.
6. FRECHETTE, V. D. *Markings on Crack Surfaces of Brittle Materials: A Suggested Unified Nomenclature* in ASTM STP 827, p. 104-109.

### Fractography of Ceramics, Overview Papers

1. RICE, R. W. *Fractographic Identification of Strength Controlling Flaws and Microstructure* in Fracture Mechanics of Ceramics, Vol. 1, R. Bradt, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1974, p. 323-345.
2. MECHOLSKY, J. J., Jr., and FREIMAN, S. W. *Determination of Fracture Mechanics Parameters Through Fractographic Analysis of Ceramics* in Fracture Mechanics of Ceramics Applied to Brittle Materials. S. Freiman, ed., ASTM STP 678, 1979, p. 136-150.
3. QUINN, G. D. *Fractographic Analysis and the Army Flexure Test Method* in Fractography of Glasses and Ceramics. Advances in Ceramics, Vol. 22, J. Varner and V. Frechette, eds., American Ceramic Society, Westerville, OH, 1988, p. 319-334.

4. RICE, R. W. *Topography of Ceramics in Surfaces and Interfaces of Glass and Ceramics*. V. Frechette, W. LaCourse, and V. Burdick, eds., Plenum Press, New York, 1974, p. 439-472.

5. RICE, R. W. *Ceramic Fracture Features, Observations, Mechanisms and Uses in Fractography of Ceramic and Metal Failures*. ASTM STP 827, 1984, p. 5-103.

6. RICHESON, D. W. *Failure Analysis, Chapter 12 in Modern Ceramic Engineering*. Marcel Dekker, New York, 1982, p. 325-375.

#### **Defects in Advanced Ceramics**

1. KIRCHNER, H., GRUVER, R., and SOTTER, W. *Characteristics of Flaws at Fracture Origins and Fracture Stress - Flaw Size Relations in Various Ceramics*. Mat. Sci. Eng., v. 22, 1976, p. 147-156.

2. BAUMGARTNER, H. and RICHESON, D. *Inclusion Effects on the Strength of Hot Pressed  $Si_3N_4$  in Fracture Mechanics of Ceramics*, Vol. 1, R. Bradt, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1974, p. 367-386.

3. GEE, M. G., and MORRELL, R. *Fracture Mechanics and Microstructures in Fracture Mechanics of Ceramics*, Vol. 8. R. Bradt, A. Evans, D. Hasselman, and F. Lange, eds., Plenum Press, New York, 1986, p. 1-22.

4. EVANS, A. G. *Structural Reliability, A Processing Dependent Phenomenon*. J. Am. Ceram. Soc., v. 65, no. 3, 1982, p. 127-139.

5. RICE, R. W. *Failure Initiation in Ceramics: Challenges of NDE and Processing*. In Preparation.

6. RICE, R. W. *Processing Induced Sources of Mechanical Failure in Ceramics in Processing of Crystalline Ceramics*. H. Palmour, R. Davis, and T. Hare, eds., Plenum Press, New York, 1978, p. 303-319.

7. RICE, R. W., MECHOLSKY, J. J., Jr., and BECHER, P. F. *The Effect of Grinding Direction on Flaw Character and Strength of Single Crystal and Polycrystalline Ceramics*. J. Mat. Sci., v. 16, 1981, p. 853-862.

8. MECHOLSKY, J. J., Jr., FREIMAN, S. W., and RICE, R. W. *Effects of Grinding on Flaw Geometry and Fracture of Glass*. J. Am. Ceram. Soc., v. 60, no. 3-4, 1977, p. 114-117.

9. RICE, R. W. *Pores as Fracture Origins in Ceramics*. J. Mat. Sci., v. 19, 1984, p. 895-914.

10. RICHESON, D. W. *Failure Analysis, Chapter 12 in Modern Ceramic Engineering*. Marcel Dekker, New York, 1982, p. 325-375.

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1. MECHOLSKY, J. J., Jr., FREIMAN, S. W., and RICE, R. W. *Fracture Surface Analysis of Ceramics*. J. Mat. Sci., v. 11, 1976, p. 1310-1319.



2. MECHOLSKY, J. J., Jr., RICE, R. W., and FREIMAN, S. W. *Prediction of Fracture Energy and Flaw Sizes in Glasses from Measurements of Mirror Size*. J. Am. Ceram. Soc., v. 57, no. 10, 1974, p. 440-443.
3. KIRCHNER, H. P., GRUVER, R. M., and SOTTER, W. A. *Fracture Stress - Mirror Size Relations for Polycrystalline Ceramics*. Phil. Mag., v. 33, no. 5, 1976, p. 775-780.
4. KIRCHNER, H. P., and CONWAY, J. C., Jr. *Fracture Mechanics of Crack Branching in Ceramics* in Fractography of Glass and Ceramics. Advances in Ceramics, Vol. 22, American Ceramic Society, Westerville, Ohio, 1988, p. 187-213.
5. MECHOLSKY, J. J., Jr., and FREIMAN, S. W. *Determination of Fracture Mechanics Parameters Through Fractographic Analysis of Ceramics* in Fracture Mechanics Applied to Brittle Materials. ASTM STP 678, S. Freiman, ed., 1979, p. 136-150.
6. KIRCHNER, H. P., and KIRCHNER, J. *Fracture Mechanics of Fracture Mirrors*. J. Am. Ceram. Soc., v. 62, no. 3-4, 1979, p. 198-202.

## APPENDIX 2. SUGGESTED FLAW NOMENCLATURE FOR STRUCTURAL CERAMICS

<u>FLAW TYPE</u>	<u>CODE</u>
<b>Intrinsic Volume Flaws:</b>	
<u>PORE:</u>  A small opening, void, interstice or channel within a consolidated solid mass or agglomerate; usually larger than atomic or molecular dimensions. (Ceram. Glossary).*  An internal cavity which may be exposed by cutting, grinding, polishing, or fracture to become a pit, pock, or hole. (F109-73).†  A discrete cavity or void in a solid materials or a cavity or void larger than the typical porosity that might be present.	P
<u>POROUS SEAM:</u>  A two-dimensional area of porosity or microporosity of higher concentration than is normally found in the matrix.	PS
<u>POROUS REGION:</u>  A three-dimensional zone of porosity or microporosity of higher concentration than is normally found in the matrix.	PR
<u>AGGLOMERATE:</u>  The clustering together of a few to many particles, whiskers, or fibers or a combination thereof, into a larger solid mass. (Ceram. Glossary).*	A
<u>INCLUSION:</u>  A foreign body from other than the normal composition enclosed in the matrix. (Ceram. Glossary)* (F109-73).†	I
<u>SECOND PHASE INHOMOGENEITY:</u>  A microstructural irregularity related to the nonuniform distribution of a second phase (e.g., an atypically large pocket of a second phase or a second phase zone of composition or crystalline phase structure different than the matrix material).	2P
<u>LARGE GRAIN(S):</u>  A single (or cluster of) unusually large grain(s).	LG

**CRACK:**

CK

A line of fracture without complete separation.  
(Ceram. Glossary)\* (F109-73).<sup>†</sup>

**Intrinsic Surface Flaws:**

**MACHINING DAMAGE:**

md

Atypical or excessively large surface microcracks or damage resulting from the machining process (e.g., striations, scratches, impact cracks).  
(Note: Small surface and subsurface damage is intrinsic to the machining damage.)

**HANDLING DAMAGE:**

hd

Scratches, chips, cracks, etc., due to the handling of the specimen.

**PIT:**

pt

A defect created by exposure to the environment (e. g., corrosion, thermal cycling, etc.).

**SURFACE VOID:**

sv

A void which is located at the surface and is a consequence of processing; i.e., surface reaction layer, as distinguished from a volume distributed flaw.

**Miscellaneous:**

**OTHER:** A defect specific to a material.

@

**?????:** An uncertain or undetermined flaw.

?

\*From the 1984 Ceramic Glossary.

<sup>†</sup>From ASTM F109-73, "Surface Imperfections on Ceramics".

@ OTHER types of flaws is up to the discretion of the user.

### APPENDIX 3. ENCYCLOPEDIA OF FLAWS

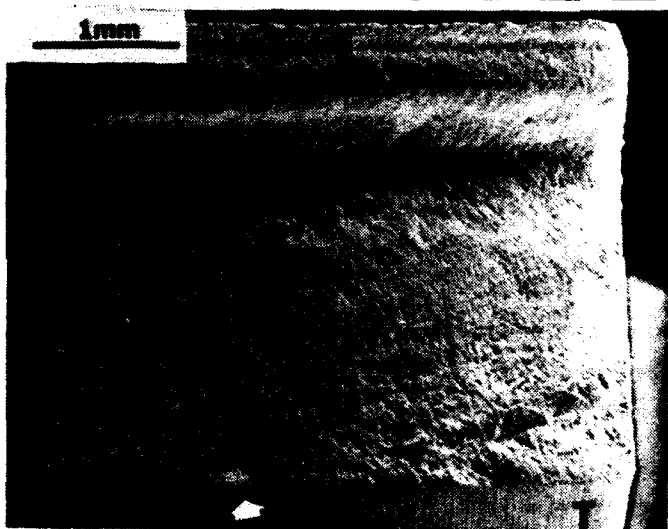


**FLAW:** PORE

**MATERIAL:** Sintered Yttria-Tetragonal Zirconia Polycrystal, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature after being exposed to  $\approx 800$  Pa water vapor pressure at 200°C for 50 hours.

**COMMENTS:**  $\sigma = 544$  MPa.

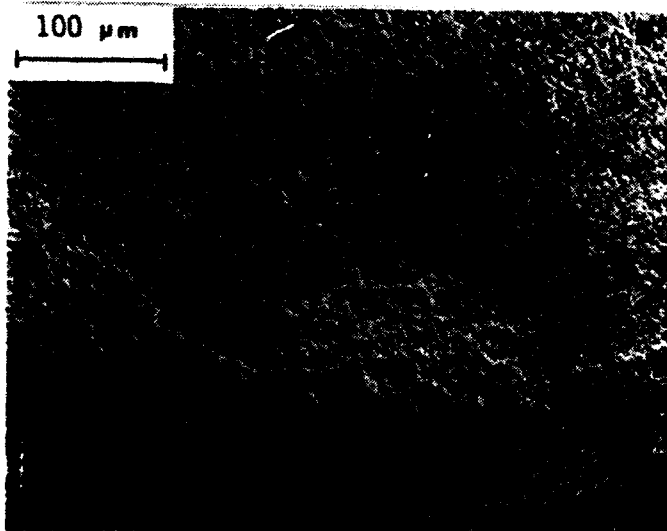


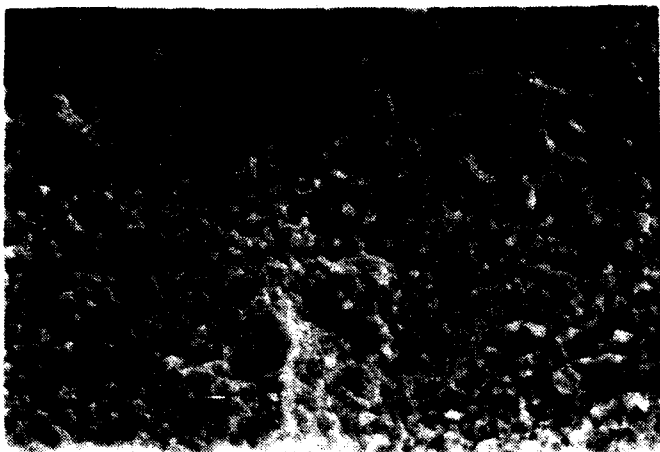
**FLAW:** POROUS SEAM

**MATERIAL:** Sintered (99.9% pure) Alumina, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 329$  MPa;  
-Photo A is from low magnification SEM analysis;  
-Photo B is from high magnification SEM analysis.



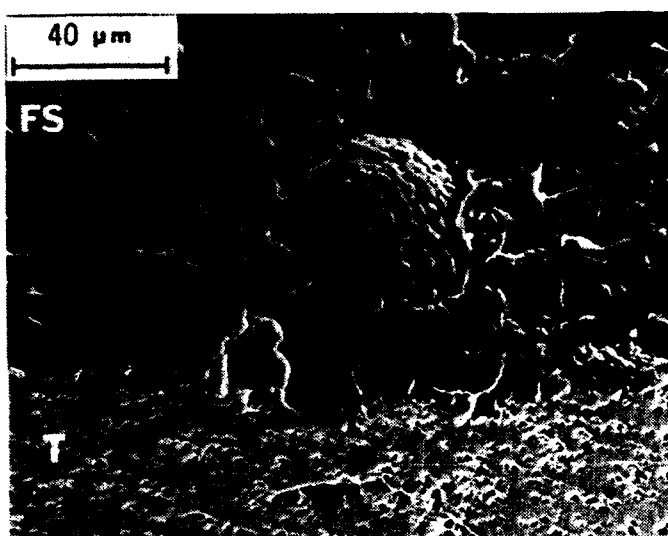


**FLAW:** POROUS REGION

**MATERIAL:** Sintered (99.9% pure) Alumina, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 419$  MPa;  
-Defect is a zone of circumferential microporosity.

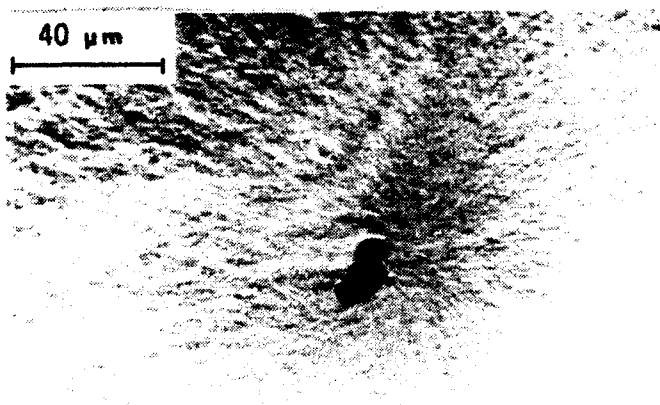


**FLAW:** AGGLOMERATE

**MATERIAL:** Sintered  $\alpha$ -Silicon Carbide, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 362$  MPa.

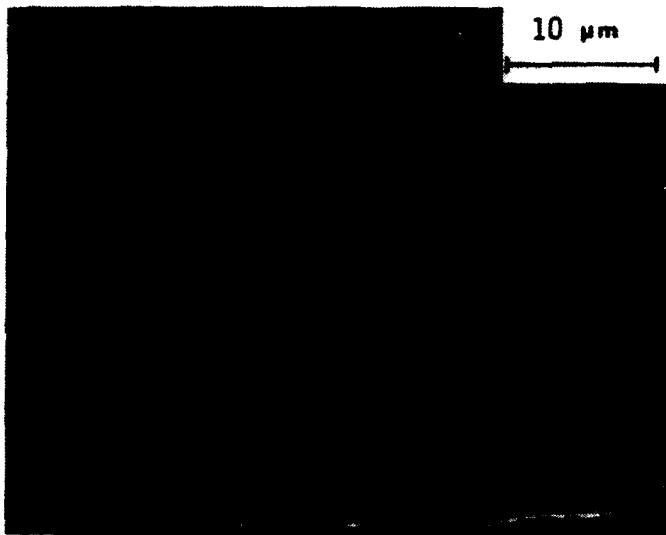


**FLAW:** INCLUSION

**MATERIAL:** Hot pressed Ytria-Tetragonal Zirconia Polycrystal, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 1029$  MPa;  
-Inclusion is elemental Silicon.

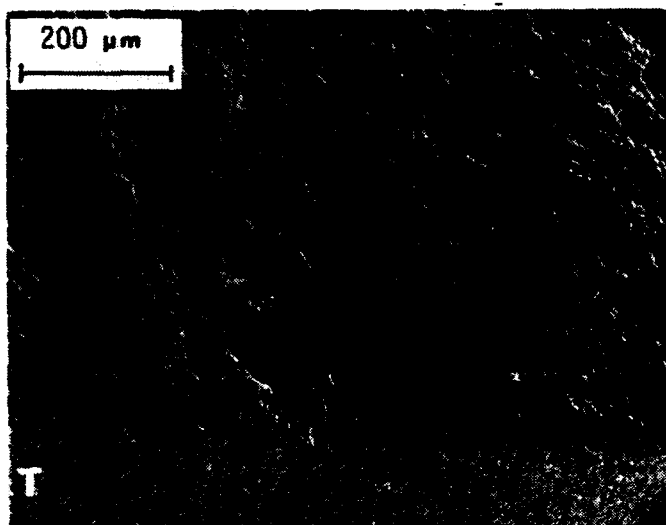


**FLAW:** SECOND PHASE  
INHOMOGENIETY

**MATERIAL:** Hot pressed Ytria-  
Tetragonal Zirconia Polycrystal,  
as-machined.

**TEST CONDITIONS:** Fast fracture  
in 4-point flexure at room  
temperature after being exposed  
to 1000°C for 500 hours.

**COMMENTS:**  $\sigma = 1033$  MPa;  
-Second Phase is elemental  
Aluminum.

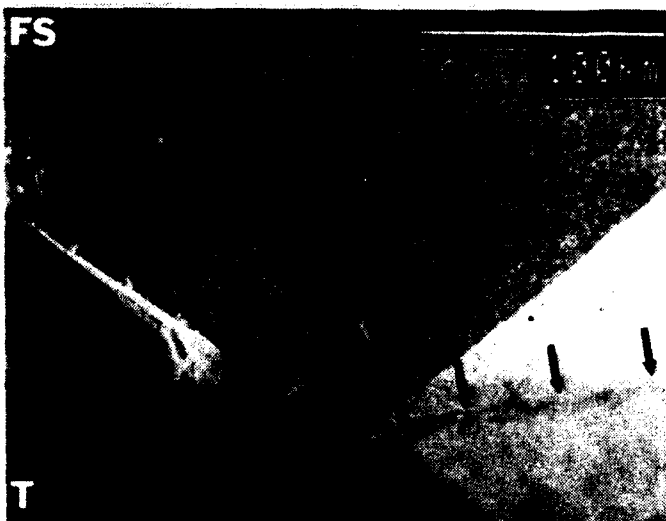


**FLAW:** LARGE GRAINS

**MATERIAL:** Siliconized Silicon  
Carbide, as-machined.

**TEST CONDITIONS:** Fast fracture  
in 4-point flexure at room  
temperature.

**COMMENTS:**  $\sigma = 169$  MPa;  
-Defect is large grains of  
Silicon Carbide.

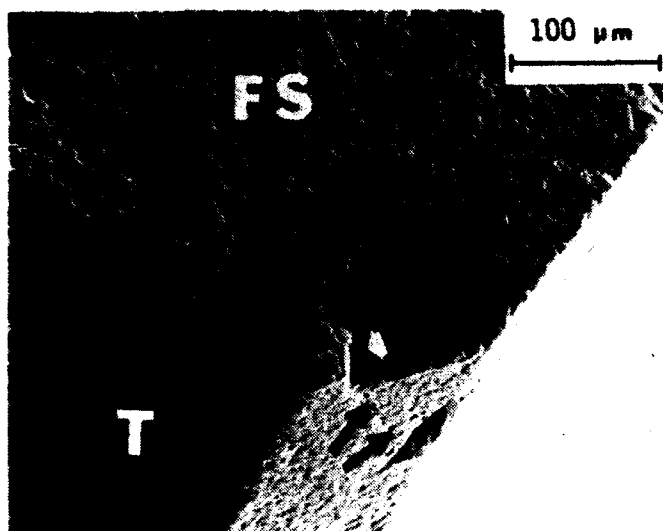


**FLAW:** CRACK

**MATERIAL:** Hot pressed Ytria-  
Tetragonal Zirconia Polycrystal,  
as-machined.

**TEST CONDITIONS:** Fast fracture  
in 4-point flexure at room  
temperature after being exposed  
to  $\approx 800$  Pa water vapor pressure  
at 400°C for 50 hours.

**COMMENTS:**  $\sigma = 514$  MPa.

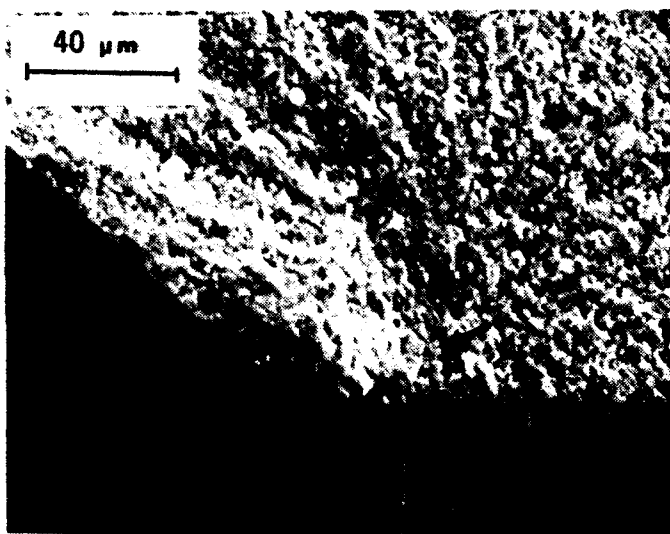


**FLAW:** MACHINING DAMAGE

**MATERIAL:** Reaction Bonded Silicon Nitride, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 264$  MPa;  
-White arrow indicate subsurface machining damage associated with the machining of the chamfer (black arrows).



**FLAW:** HANDLING DAMAGE

**MATERIAL:** Sintered Sialon with Ytria and Alumina additions, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 595$  MPa;  
-Scratch on the tensile surface.

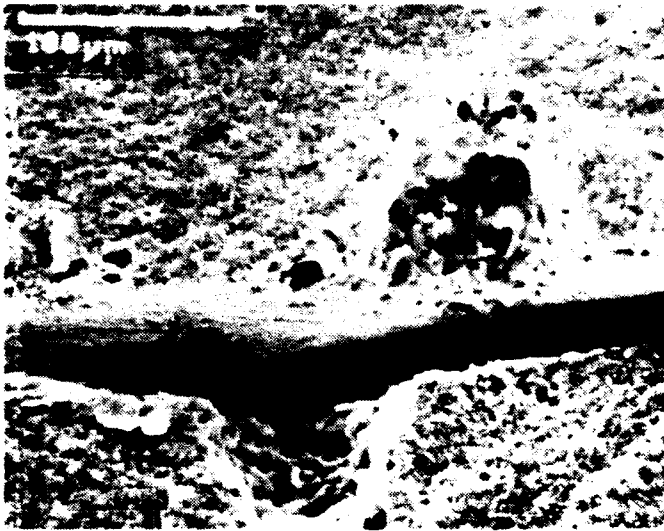


**FLAW:** PIT

**MATERIAL:** Hot pressed Silicon Nitride with Magnesia additions, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature after being exposed to 1000°C for 500 hours.

**COMMENTS:**  $\sigma = 598$  MPa;  
-Pits formed due to oxidation.



**FLAW:** SURFACE VOID

**MATERIAL:** Injection Molded and Sintered Silicon Nitride with Yttria and Alumina additions, as-fired.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 472$  MPa;  
-Photo shows both halves of the fracture surface.



**FLAW:** OTHER

**MATERIAL:** Siliconized Silicon Carbide, as-machined.

**TEST CONDITIONS:** Fast fracture in 4-point flexure at room temperature.

**COMMENTS:**  $\sigma = 282$  MPa;  
-Flaw is a vein of elemental Silicon.



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